NAS CR-135021
TRW ER-7849

TUNGSTEN WIRE-NICKEL BASE ALLOY COMPOSITE DEVELOPMENT

BY

W.D.BRENTNALL
D.J.MORACZ

TRW EQUIPMENT

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
NASA Lewis Research Center

Contract NAS 3-17816

D. W. Petrasek, Project Manager

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA CR-135021		

4. Title and Subtitle	5. Report Date
Tungsten Wire-Nickel Base Alloy Composite Development	March 1976
	6. Performing Organization Code
7. Author(s)	8. Performing Organization Report No.
W. D. Brentnall and D. J. Moracz	ER-7849
	10. Work Unit No.
9. Performing Organization Name and Address	11. Contract or Grant No.
TRW Inc. 23555 Euclid Avenue Cleveland, Ohio 44117	NAS 3-17816
	13. Type of Report and Period Covered
12. Sponsoring Agency Name and Address	Contractor Report June 1, 1974-February 29,1976
National Aeronautics and Space Administration Washington, D. C. 20546	14. Sponsoring Agency Code

15. Supplementary Notes

Project Manager, D. W. Petrasek
NASA/Lewis Research Center
21000 Brookpark
Cleveland, Ohio 44117

16. Abstract

Further development and evaluation of refractory wire reinforced nickel-base alloy composites is described. Emphasis was placed on evaluating thermal fatigue resistance as a function of matrix alloy composition, fabrication variables and reinforcement level and distribution. Tests for up to 1000 cycles were performed and the best system identified in this current work was 50v/o W/NiCrAlY. Improved resistance to thermal fatigue damage would be anticipated for specimens fabricated via optimized processing schedules. Other properties investigated included 1093°C (2000°F)stress rupture strength, impact resistance and static air oxidation. A composite consisting of 30v/o W-Hf-C alloy fibers in a NiCrAlY alloy matrix was shown to have a 100-hour stress rupture strength at 1093°C (2000°F) of 365 MN/M^2 (53 ksi) or a specific strength advantage of about 3:1 over typical D.S. eutectics.

17. Key Words (Suggested by Author(s))		18. Distribution Statement
Composites	Stress Rupture	Unclassified - Unlimited
Turbine Blades	Impact Resistance	
Nickel Alloy	Oxidation	
Tungsten Wire		
Thermal Fatigue		

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	-	$3.00

*For sale by the National Technical Information Service, Springfield, Virginia 22161

FORWORD

The work described in this report was performed in the Materials Technology Laboratory of TRW Inc, under sponsorship of the National Aeronautics and Space Administration, Contract NAS 3-17816. The principal investigator was Mr. W. D. Brentnall with technical contributions by Mr. D. J. Moracz. Composite fabrication and some of the mechanical property evaluations were performed by Mr. M. J. Cooney. The NASA Technical Manager was Mr. D. W. Petrasek.

The TRW Report Number is ER-7849.

ABSTRACT

Further development and evaluation of refractory wire reinforced nickel-base alloy composites is described. Emphasis was placed on evaluating thermal fatigue resistance as a function of matrix alloy composition, fabrication variables and reinforcement level and distribution. Tests for up to 1000 cycles were performed and the best system identified in this current work was 50v/o W/NiCrAlY. Improved resistance to thermal fatigue damage would be anticipated for specimens fabricated via optimized processing schedules. Other properties investigated included 1093°C (2000°F) stress rupture strength, impact resistance and static air oxidation. A composite consisting of 30v/o W-Hf-C alloy fibers in an NiCrAlY alloy matrix was shown to have a 100-hour stress rupture strength at 1093°C (2000°F) of 365 MN/m^2 (53 ksi) or a specific strength advantage of about 3:1 over typical D.S. eutectics.

TABLE OF CONTENTS

		Page No.
1.0	SUMMARY	1
2.0	INTRODUCTION	2
3.0	PROGRAM PLAN	3
	TASK I - Thermal Cycle Resistant Composite Development	3
	Selection of Materials	3
	Fabrication Methods	3
	Thermal Fatigue Screening Studies	4
	Advanced Thermal Fatigue Characterization Studies	4
	TASK II - Matrix Development & Composite Characterization	5
	Selection of Materials	5
	Evaluation of Composite Properties	5
	Material Identification Code	6
4.0	RESULTS AND DISCUSSION	7
	Material Procurement	7
	Thermal Fatigue Screening Evaluations	9
	Specimen Design	9
	TRW Thermal Fatigue Screening Studies	11
	Results of Thermal Fatigue Screening Studies	17
	Advanced Thermal Fatigue Characterization Studies	32
	Composite Characterization Studies	36
	Systems Selection	36
	Stress Rupture	36
	Miniature Izod Impact Tests	38
	Static Air Oxidation	46
5.0	CONCLUSIONS AND RECOMMENDATIONS	53
6.0	REFERENCES	55
7.0	APPENDIX - Vendor Certification	56
8.0	DISTRIBUTION LIST	57

1.0 SUMMARY

The objective of this program was to continue the development and evaluation of refractory wire reinforced nickel base alloy composites which are designed for advanced gas turbine engine blades capable of operating at temperatures up to 1093°C (2000°F). A variety of nickel-base alloy matrices with 218 W wire reinforcement were investigated and the evaluations included thermal fatigue, stress rupture, impact and static oxidation tests. A major goal was the development of systems capable of withstanding, without significant damage, 1000 thermal cycles representative of gas turbine engine operating cycles. Thermal cycling was observed to produce a variety of effects including dimensional instability, warpage, delamination, matrix debonding and matrix microcracking. Data obtained on these and other systems indicate that these effects may be controlled by a proper selection of matrix strength, fiber strength, and volume fraction and distribution of the reinforcement. Specimens fabricated with the NiCrAlY matrix had the best thermal fatigue resistance but developed internal microcracking after 100 thermal cycles. It was felt that development of optimized fabrication processes would solve this problem based on metallographic observations and successful test data obtained on W/FeCrAlY composites in other work.

Stress rupture strengths were shown to be at least as high as theoretical predictions, based on published data for the tungsten fibers. The 100-hour 1093°C (2000°F) stress rupture strength of a composite containing 30 volume percent of an advanced W-Hf-C fiber reinforcement was determined as about 365 MN/m^2 (53 ksi) which translates to a specific strength advantage of about 3:1 over typical D.S. eutectics.

Notched impact tests identified a DBTT range between 150° and 300°C below which fracture was matrix controlled and above which fracture was controlled by the fibers. The notched impact values were in the range defined as adequate for turbine blade applications. Oxidation rates at 1093°C (2000°F) corresponded to anticipated rates for the respective matrix alloy compositions.

2.0 INTRODUCTION

The purpose of this program was to continue development of nickel-base alloy matrix composites reinforced with refractory metal fibers which have high potential for advanced aircraft engine applications such as turbine blades. Of major concern in this program was the composite's response to thermal cycling, and much of the effort was devoted towards developing a composite having good thermal fatigue properties. A specific fiber-matrix combination was selected for preliminary characterization of the properties necessary to design a turbine blade.

Combinations of refractory-metal fibers and superalloys in the form of composites have been made by NASA and others and showed promise from stress-rupture and impact data that have been generated. For example, programs have been conducted at NASA-Lewis Research Center[1-4] which have demonstrated that composites could be produced having superior stress-rupture properties compared to conventional superalloys at temperatures of 1093° and 1204°C (2000° and 2200°F). Additionally, it was shown that by careful fabrication techniques and selection of compatible matrix alloys, Ni alloy/W composites could be produced having adequate impact properties for turbine blade or vane applications. This work identified the relationships of matrix composition, compatibility (diffusional stability), elevated temperature strength and impact toughness in these systems.

A recent NASA-sponsored program at TRW[5] showed that advanced fabrication methods could be used to produce composites with stress-rupture properties at least as good as those developed in the previous work. Further, the indications were that improved ductility, leading to better impact and thermal fatigue properties, may be achieved by virtue of increased matrix deformation in the process compared to the slip casting/hot isostatic pressing method used previously. The TRW diffusion bonding process may be readily applied to the fabrication of solid air foil shapes incorporating cross plied or variable volume fraction, filament distributions, or even to hollow airfoil shapes.

The experimental program was conducted in two tasks: Task I - Thermal Cycle Resistant Composite Material Development, and Task II - Matrix Develoment and Composite Characterization.

3.0 PROGRAM PLAN

The overall program plan is summarized in the following paragraphs:

TASK I - Thermal Cycle Resistant Composite Development

The primary objective of Task I was to define the optimum combination of matrix alloy, fiber volume fraction and fiber distribution, in terms of the materials' resistance to thermal fatigue damage. It was planned to evaluate four matrix types which would represent variations in composition and starting condition (powder or wrought). The selected reinforcement was 0.038 cm (0.015 in) diameter 218 CS tungsten wire. Test specimen panels were to be fabricated by hot pressing precollimated fibers between layers of superalloy powder cloth or superalloy sheet material to make panels having the desired play configurations.

Selection of Materials

a. Fiber Reinforcement

The fiber reinforcement selected for Task I evaluations was the General Electric type 218 CS tungsten lamp filament wire of 0.038 cm (0.015 in) diameter. This material had been well characterized in previous NASA investigations [1&3] and on the previous NASA/TRW program [4] and represented a readily available, relatively low cost reinforcement.

b. Matrix Materials

Important considerations in the selection of matrix alloys included oxidation/corrosion resistance, compatibility with the fibers, thermal fatigue properties, ductility and fabricability. Data previously obtained by NASA and by TRW on Contract NAS 316756 were directly applied in the evaluation of potential matrix alloys. The selected matrix alloys and conditions are:

56Ni-25W-15CR-2Ti-2Al (Alloy 111) - Powder;
74.6Ni-20Cr-5Al-0.4Y (NiCrAlY) - Powder and Sheet;
Ni-12Cr-10Co-6W-3Mo-1.5Ta-4.6Al-3Ti-0.3C (2-1DA) - Powder and Sheet.

The powder alloy had a particle size of -325 to -500 mesh and were prepared to insure minimum contamination. The total oxygen level was to be less than 200 ppm, carbon less than 0.02 wt percent, and sulphur plus phosphorous less than 0.005 wt percent. The alloy sheets materials were procured in 0.051 cm and 0.318 cm (0.020 inch and 0.125 inch gage thicknesses).

Fabrication Methods

Thermal fatigue test specimens were to be fabricated by the diffusion bonding process described in NASA CR-134664 [5]. Typical diffusion bonding conditions were: pressure - 10-30 ksi; temperature - 982-1204°C (1800-2200°F); and time - 5-30 minutes. Other processes that might result in a more heavily worked matrix (particularly for the powder matrix systems), such as hot rolling, were to be investigated in Task I.

Thermal Fatigue Screening Studies

Initially screening studies were to be conducted to investigate the effect of a number of variables on the composite's resistance to rapid thermal cycling. The test procedure used is described in Section 4.0. Briefly, the specimen is heated by direct resistance heating and subjected to a programmed heating and cooling cycle. Preliminary evaluations were to be restricted to 100 cycle exposures between 38°-1093°C (100°-2000°F). Evaluations included visual inspections, measurements of geometric distortion and dimensional changes, metallographic and scanning electron microscopy investigations.

a. Specimen Geometry Effect

The effect of specimen geometry on thermal fatigue results were to be investigated for the alloy 111 matrix with 50 and 35 or 65 volume percent reinforcement levels (volume fraction to depend on material behavior). Two simple geometry specimen designs were selected for investigation; a square specimen .64 x .64 x 12.7 cm (.25 x .25 x 5 inch) and a sheet type specimen .32 x .64 x 12.7 cm (.125 x .25 x 5 inch).

b. Matrix Alloy/Volume Fraction Relationships

Determination of the effect of matrix alloy strength and ductility on the "critical" volume fraction reinforcement corresponding to minimum thermal fatigue damage was the goal of this sub-task. Reinforcement levels - 50 and 35 or 65 volume percent - were to be investigated for each matrix using one selected test specimen geometry.

c. Effect of Cross Plying

The effect of cross plying on dimensional stability during thermal cycling was to be investigated for one matrix alloy and one volume fraction level. A balanced cross ply configuration such as +45, -45, 0, 0, -45, +45 or +15, -15, 0, 0, -15, +15 was to be selected for thermal cycling tests.

d. Matrix Alloy Condition

Comparison of resistance to thermal cycling of composites fabricated from pre-alloyed powder matrix and wrought sheet was to be investigated for 2-1DA matrix and NiCrAlY matrices. Two levels of reinforcement - 50 and 35 or 65 volume percent - were selected for investigation.

e. Matrix Conditioning

The potential of matrix conditioning or ductility enhancement by thermal-mechanical processing was investigated for one matrix alloy-fiber combination.

f. External Load Effects

The effect of an applied load on thermal fatigue resistance was to be investigated for one matrix alloy-fiber combination.

Advanced Thermal Fatigue Characterization Studies

To demonstrate the thermal fatigue resistance of the composite materials for a greater number of cycles several composite systems were tested for 1000 cycles

between 427°-1093°C (800°-2000°F). The selected composite systems were comprised of two matrix alloys each with two fiber reinforcement levels.

TASK II - Matrix Development and Composite Characterization

The primary objective of this task was to optimize the matrix material composition to impart the best overall properties to the composite. Two nickel-base alloys were to be investigated as candidate matrix materials. Selection criterial included compatibility with tungsten wire, oxidation resistance, thermal fatigue resistance, impact resistance and elevated temperature strength. Composite specimens were to be finally fabricated for evaluation by stress-rupture, impact and oxidation studies.

Selection of Materials

a. Fiber Reinforcement

The selected fiber reinforcement was General Electric type 218 CS tungsten lamp filament wire of 0.038 cm (0.015 inch) diameter. It was also planned to use W-Hf-C wire of the same diameter for some composite property determinations.

b. Matrix Materials

Two nickel-base alloy matrix materials were to be selected from Task I based on their expected compatibility with tungsten wire, oxidation resistance, thermal fatigue resistance, impact resistance and elevated strength properties.

Evaluation of Composite Properties

Composite specimens were to be fabricated by the diffusion bonding process as described in NASA CR-134664[5]. Composite specimens were to be fabricated containing fiber contents between 35 and 65 volume percent for characterization by the evaluations listed below.

a. Stress-Rupture Tests

- Test Range: Stress-to-rupture in 100 hours for 35-65 volume percent fiber content composites to be determined at 1093°C (2000°F) in air.

- Evaluation: Constant load stress rupture tests. Fracture and cross sections of specimens were to be examined after failure. The actual volume percent fiber content of the tested specimens were to be determined.

b. Impact Tests

- Test Range: Impact tests at room temperature, 149°C (300°F), 260°C (500°F) and 371°C (700°F).

- Evaluation: Miniature Izod impact tests were to be used. At least two (2) specimens of each General Electric 218 CS tungsten fiber-matrix combinations (35 to 65 volume percent fiber content) to be tested at each temeprature. Fracture surfaces and cross sections of specimens to be examined after failure. The actual fiber content of tested specimens was to be determined.

c. Oxidation Tests

- Test Range: 1093°C (2000°F).

- Evaluation: Static air oxidation tests to be made at 1093°C (2000°F). Continuous weight-gain/weight-loss measurements on at least two (2) specimens of each fiber-matrix combinations (35 to 65 volume percent fiber content) for times up to 100 hours. Evaluations to be by metallographic examinations in the longitudinal and transverse direction of the specimens.

Material Identification Code

For ease of discussion, the following reinforcement and matrix identification code was adopted for this program.

Reinforcement

The letter W is used to identify General Electric Type 218 CS tungsten lamp filament wire of 0.038 cm (0.015 inch) diameter. The volume percent (v/o) of W in the composite is identified as, for example, 35v/o W, 50 v/o W, or 65v/o W.

Matrix Materials

Code	Composition	Condition
Ni/1	56Ni- 25W-15Cr-2Ti-2Al	Powder
Ni/2	74.6Ni-20Cr-5Al-0.4Y	Powder
Ni/3	Ni-12Cr-10Co-6W-3Mo-1.5Ta-4.6Al-3Ti-0.3C	Powder
Ni/4	Ni-12Cr-10Co-6W-3Mo-1.5Ta-4.6Al-3Ti-0.3C	Sheet
Ni/5	74.6Ni-16.5Cr-5Al-0.2Y	Sheet

Composite

First part reinforcement and second part matrix, e.g., 35v/o W-Ni/2.

4.0 RESULTS AND DISCUSSION

The results of these investigations are presented and discussed under the major headings of Material Procurement, Thermal Fatigue and Mechanical Property Evaluations.

Material Procurement

Program materials and vendor sources are listed in Table I. The reinforcing fiber used in Task I evaluations was 0.038 cm (0.015 in) diameter GE type 218 CS tungsten lamp filament wire. This material has been well characterized in other NASA programs[1,3] and was used to fabricate composites in the previous NASA/TRW program. No problems have been experienced in subsequent collimation by drum winding of this wire which is supplied on 4-inch diameter spools under General Electric's standard straightness tolerance.

For some of the Task II stress rupture and thermal fatigue evaluations, composite specimens were fabricated using the high strength W-Hf-C alloy fiber material of the same diameter. This material was supplied by NASA and was fabricated, under contract, by Westinghouse. Potential problems associated with lack of straightness (curvature, kinking), were anticipated during subsequent fiber collimation, but were not in fact experienced and the "windability" of this material was as good as that of the 218 CS wire.

Nickel alloy ingots were supplied by TRW Metals for subsequent conversion to powder by Homogenous Metals Inc. Ingots weighing approximately 50 lbs were prepared by vacuum induction melting and submitted for conversion to powder by a soluble hydrogen gas technique. In the case of the Ni-20Cr-5Al-0.4Y alloy, the yttrium was added just prior to powder conversion to minimize loss. The powders were screened into three size classifications, -20+100, -100+500 and -500 mesh with the majority being in the (desired) -100+500 mesh range. Impurity level specifications were: O-200ppm max,C-0.02% max, S+P-0.005% max.

The 2-1Da sheet was obtained from Universal Cyclops in thicknesses of 0.61, 0.42, 0.317, and .051 cm (.242, .165, .125, and .020 inch). The vendor certification sheet is shown in the appendix.

Some problems were experienced in obtaining NiCrAlY alloy in sheet form. Several sources were contacted with respect to the purchase of NiCrAlY sheet but TRW was advised that current stocks of this alloy had been depleted and there were no plans to roll more material. At that time, since the program requirements were relatively small, no interest was shown by the vendors to make a special heat. It was decided, therefore, to purchase cast plates from TRW Metals Plant and attempt to roll the material in-house. This approach was later abandoned because of the difficulty of forging or rolling this coarse grained material. Later in the program, a small quantity of NiCrAlY alloy sheet was supplied by NASA (Ni, 16.5Cr, 5Al, 0.2Y, slightly different composition).

TABLE I

Program Materials and Vendors

Item	Vendor
Tungsten Wire 218 CS	GE
Tungsten Wire W-Hf-C Alloy	Westinghouse/NASA
Nickel Alloy Ingots	TRW Metals
Ni-25W-15Cr-2Ti-2Al	
Ni-20Cr-5Al-0.4Y *	
Powder Conversion Service	HMI
Nickel Alloy Sheet	Universal Cyclops
Ni-12Cr-10Co-6W-3Mo-1.5Ta	
4.6Al-3Ti-0.3C	
Nickel Alloy Cast Bar	TRW Metals
Ni-20Cr-5Al-0.4Y	
Nickel Alloy Sheet	NASA
Ni-16.5Cr-5Al-0.2Y	

* Yttrium added to melt immediately prior to powder conversion.

Thermal Fatigue Screening Evaluations

The objective of this phase of the program was to evaluate the effects of matrix alloy composition, starting condition (powder or wrought sheet), volume fraction reinforcement and the influence of an applied tensile load during thermal cycling, on thermal fatigue resistance. These screening studies were performed using the TRW Gilmore testing facility which uses direct resistance heating of the specimens and has additional capability of pre-programmed loading cycles. Of particular concern were previously noted effects such as development of internal cracks and delamination, specimen distortion and negative creep or shrinkage ("ratchetting") as a consequence of differential thermal strains developed between matrix and fibers.

Specimen Design

It was desirable to have a relatively inexpensive specimen design for these screening tests, which would avoid some of the problems previously encountered such as warpage due to the presence of relatively large unbalanced volumes of unreinforced matrix and delamination at dissimilar cladding/matrix interfaces. Most of the problems stemmed from the use of processing methods which were still in a stage of development. Specimens for the Gilmore facility could have edge-exposed fibers since the test could be conducted in an inert atmosphere. Following some preliminary tests, the specimen selected for the TRW Gilmore thermal fatigue test was a simple sheet specimen (see Figure 1) machined directly from flat panels with the dimensions 0.23-.32 x .64 x .12.7 cm (.090-.125 x .25 x .5 inch) with 5-7 plies depending on volume fraction reinforcement level.

Based on the results of the previous work, it was not felt that a fabrication optimization phase for each of the different matrix compositions and volume fraction reinforcement levels was necessary and hot pressing conditions of 1093°C (2000°F)20 ksi for 30 minutes were thought to be adequate for all but the strongest sheet alloys. As the work progressed, however, it became evident that even with the alloys in powder form, the strength differences at 1093°C were significant and resulted in varying degrees of densification. Many of the effects observed in the thermal fatigue screening evaluations could be attributed to lack of densification or poor interparticle bonding.

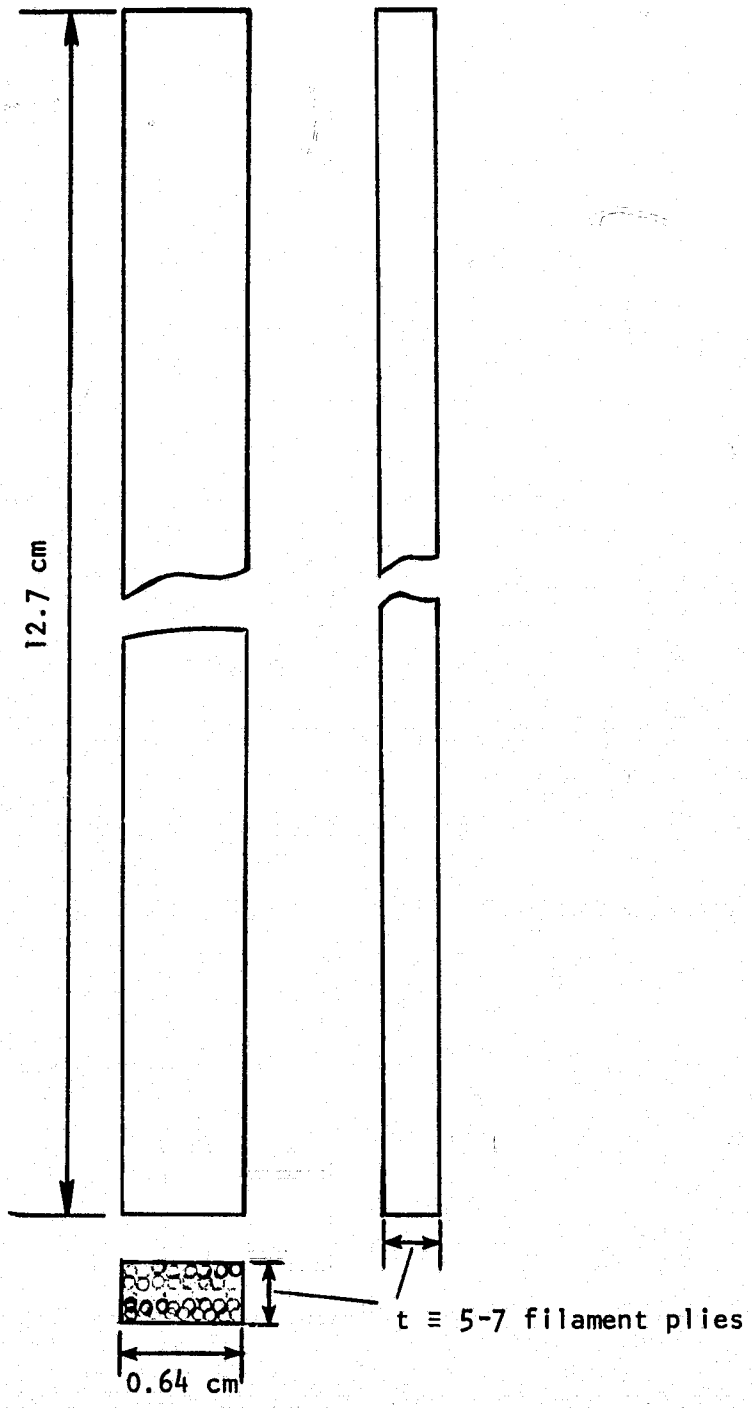


Figure 1. TRW Thermal Fatigue Specimen.

Typical as-fabricated* microstructures are shown in Figures 2 and 3. Specimens with the Ni/2 alloy matrix (NiCrAlY) were well consolidated but the outline of original powder particles could be resolved in some areas. As shown in the lower micrograph, the fiber/matrix reaction zone was barely resolvable. The specimens with the other two matrices were not so well consolidated and representative defects are shown in Figure 3. Angular shaped voids, located mostly between fibers, were observed in the W-Ni/1 (25 W alloy) system whereas most of the unconsolidated matrix in the W-Ni/3 (2-1DA) system was adjacent to the fibers and spherical powder particles could be distinguished.

TRW Thermal Fatigue Screening Studies

Test Procedure

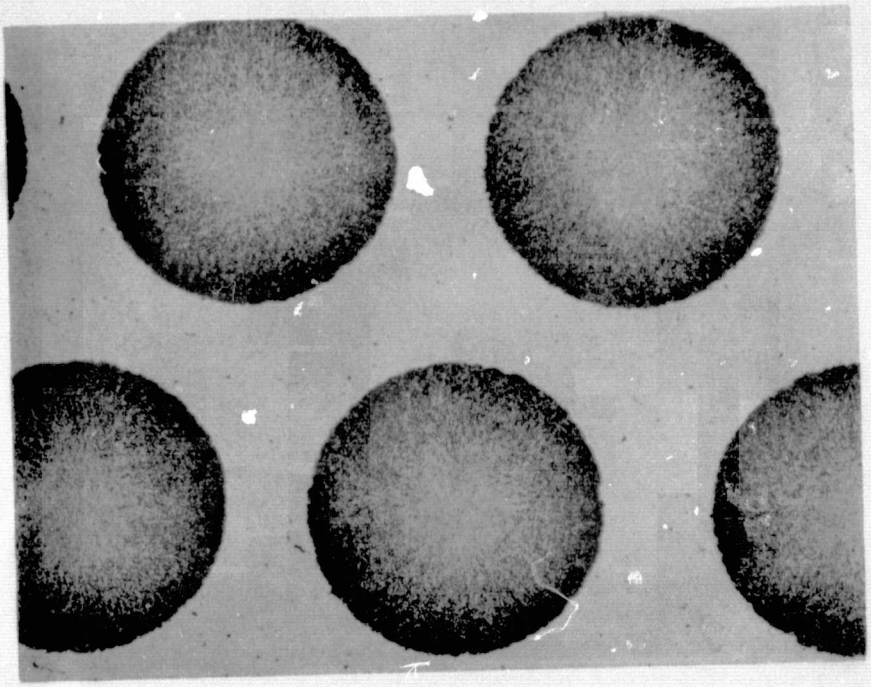
Figure 4 shows an overall view of the Gilmore test facility and Figure 5 shows a specimen mounted in the test chamber with thermocouples attached. In this test, the specimens are held in water cooled grips and direct resistance heated. In practice, there is virtually no limit to the heating rate but the maximum cooling rate is dependent upon heat removal to the cooled grips and to the surrounding environment and is therefore somewhat dependent upon specimen geometry and surrounding atmosphere. The desired heating and cooling cycle is pre-programmed. Specimens may also be subjected to superimposed, programmed load cycles through a servo-actuated hydraulic load system.

In the current tests, specimens were thermally cycled under zero load conditions or with an applied constant tensile load. Because of edge-exposed fibers, the tests were conducted in argon atmosphere; the chamber first being evacuated to about 10^{-4} torr and backfilled with argon until a constant flow rate of 4 ft^3/hour was attained. Two types of thermal cycle were used as shown in Figure 6. In the initial characterization tests, the goal was for 100 cycles between room temperature and 1093°C (2000°F) with a total cycle time of about 5 minutes.

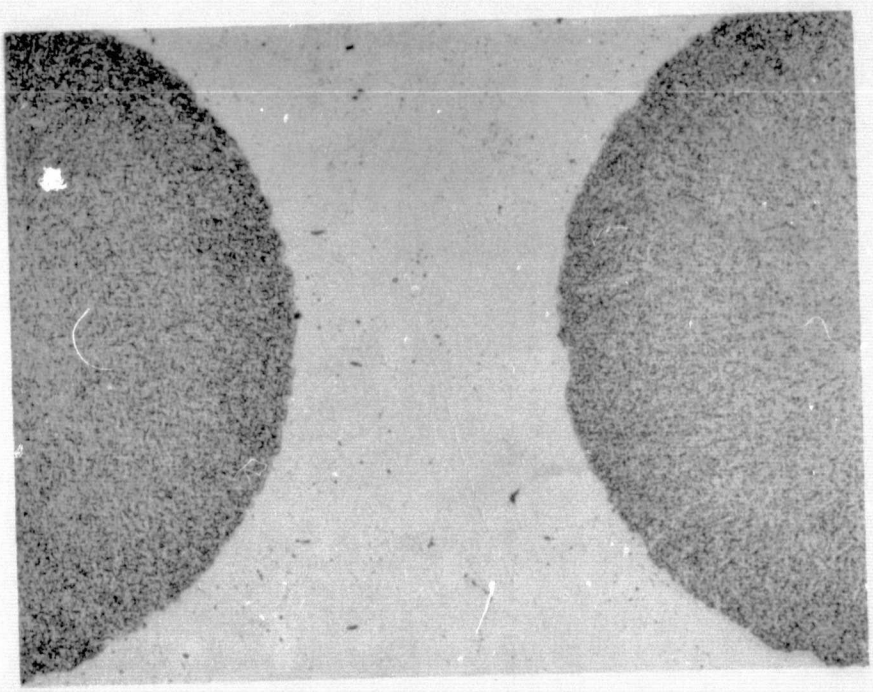
The heating rate was essentially linear to the peak temperature and equivalent to about 18C°/sec (33F°/sec) and the maximum cooling rate down to 750°F (≈400°C) was about 11C°/sec (20F°/sec). The cycle time between 399° and 1093°C (750° and 2000°F) was about 2.5 minutes, similar to the cycle used by Breinan et al[6]. It is apparent from Figure 6 that over 40% of the cycle time was due to cooling from about 450°C (800°F) to room temperature. A shorter (approximately 3 minutes) cycle of 427°-1093°C (800°-2000°F) was used for the 1000-cycle tests. A typical temperature gradient along a specimen at T_{max} as determined by thermocouples welded to the specimen surface is also shown in Figure 6. The temperature was essentially constant over the center 2.5 cm (1 inch) and there was a very large temperature gradient between the end of the water cooled grips and a point

---

* Sectioned from within the water cooled grip areaoof tested samples.

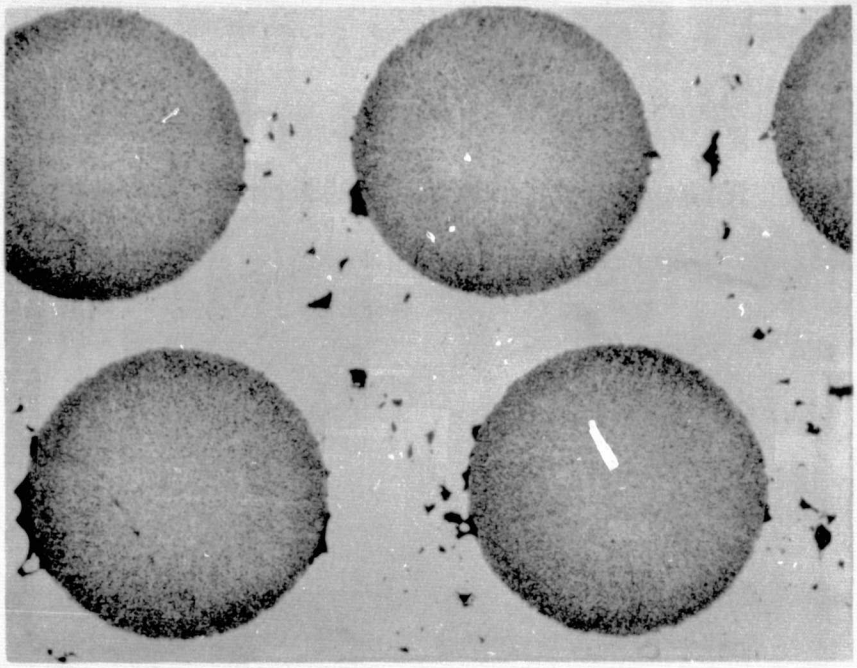


100X

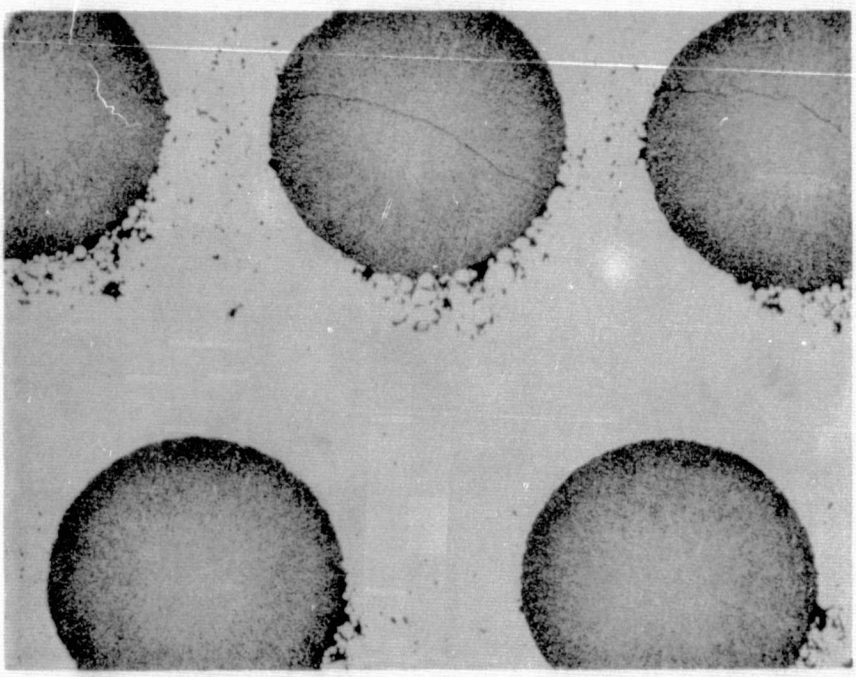


250X

Figure 2. Cross Sections of As-Fabricated 50v/o W-Ni/2 (NiCrAlY) Thermal Fatigue Specimen.



50v/o W-Ni/1 (25W Alloy) 100X



35v/o W-Ni/3 (2-1DA Powder) 100X

Figure 3. Cross Sections of As-Fabricated Thermal Fatigue Specimens.

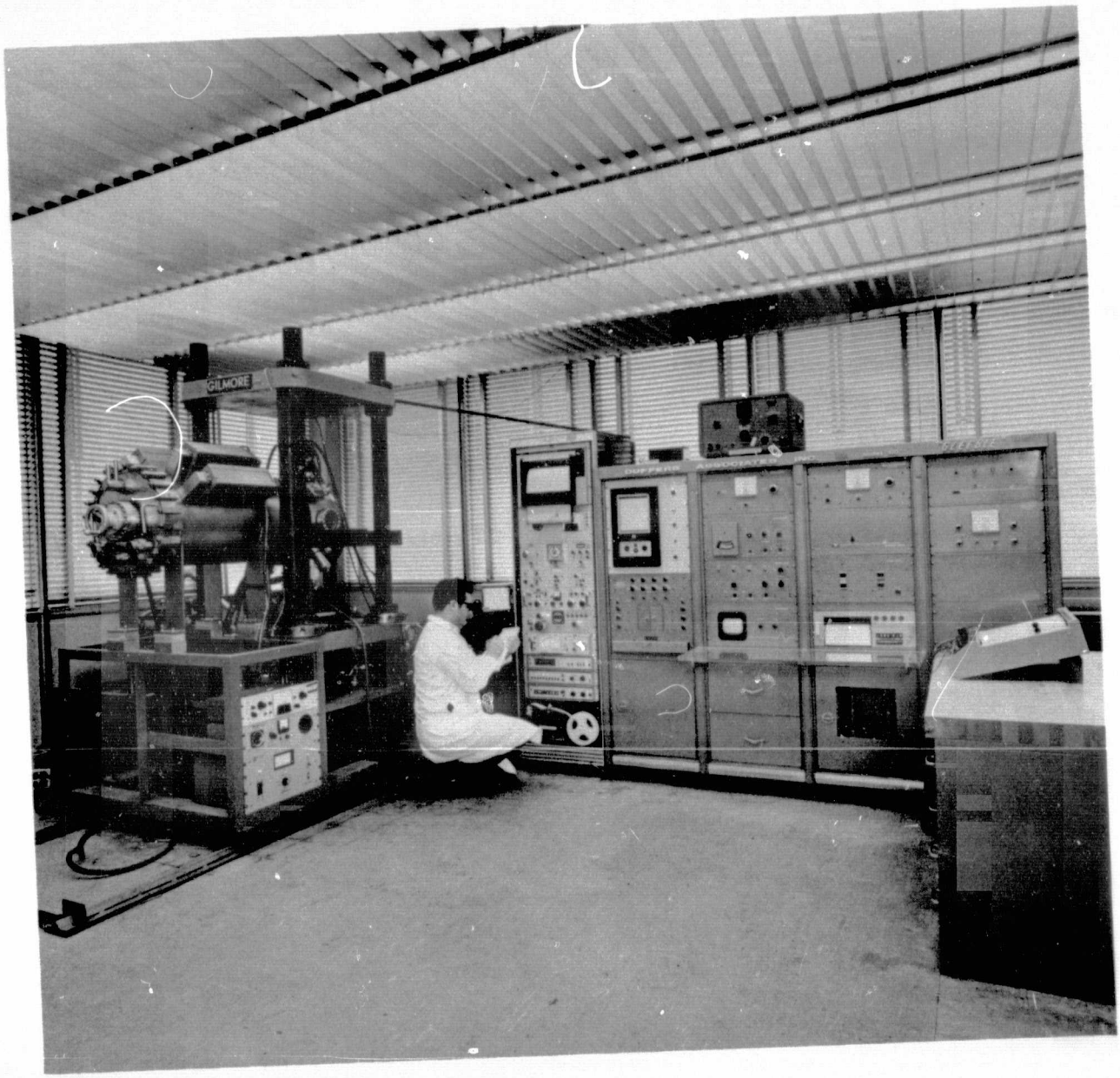


Figure 4. TRW's Gilmore Universal Testing Machine.

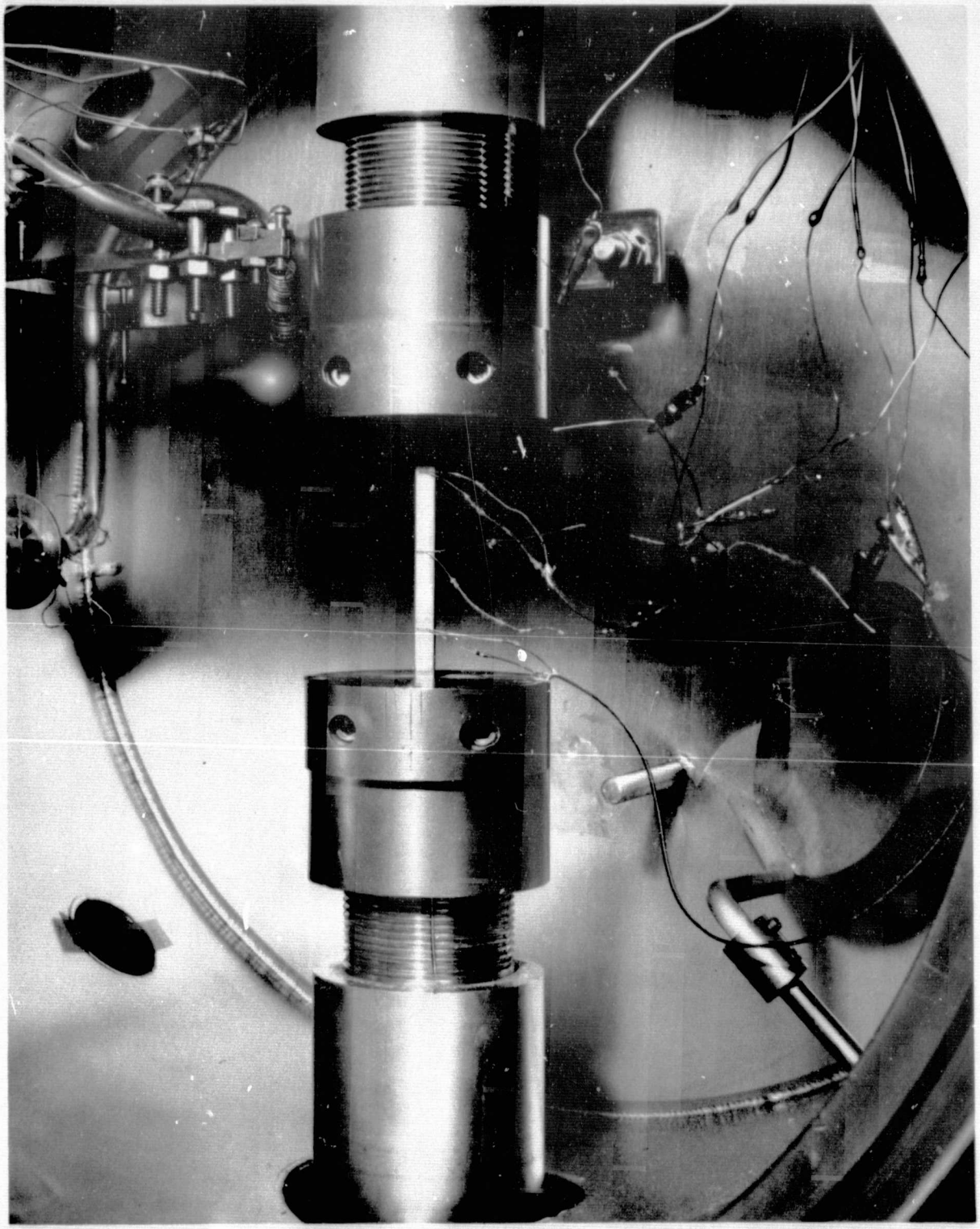


Figure 5. Close-Up View of Thermal Fatigue Specimen Mounted Between Water-Cooled Grips in the Test Chamber. Three Thermocouples Are Welded on Specimen.

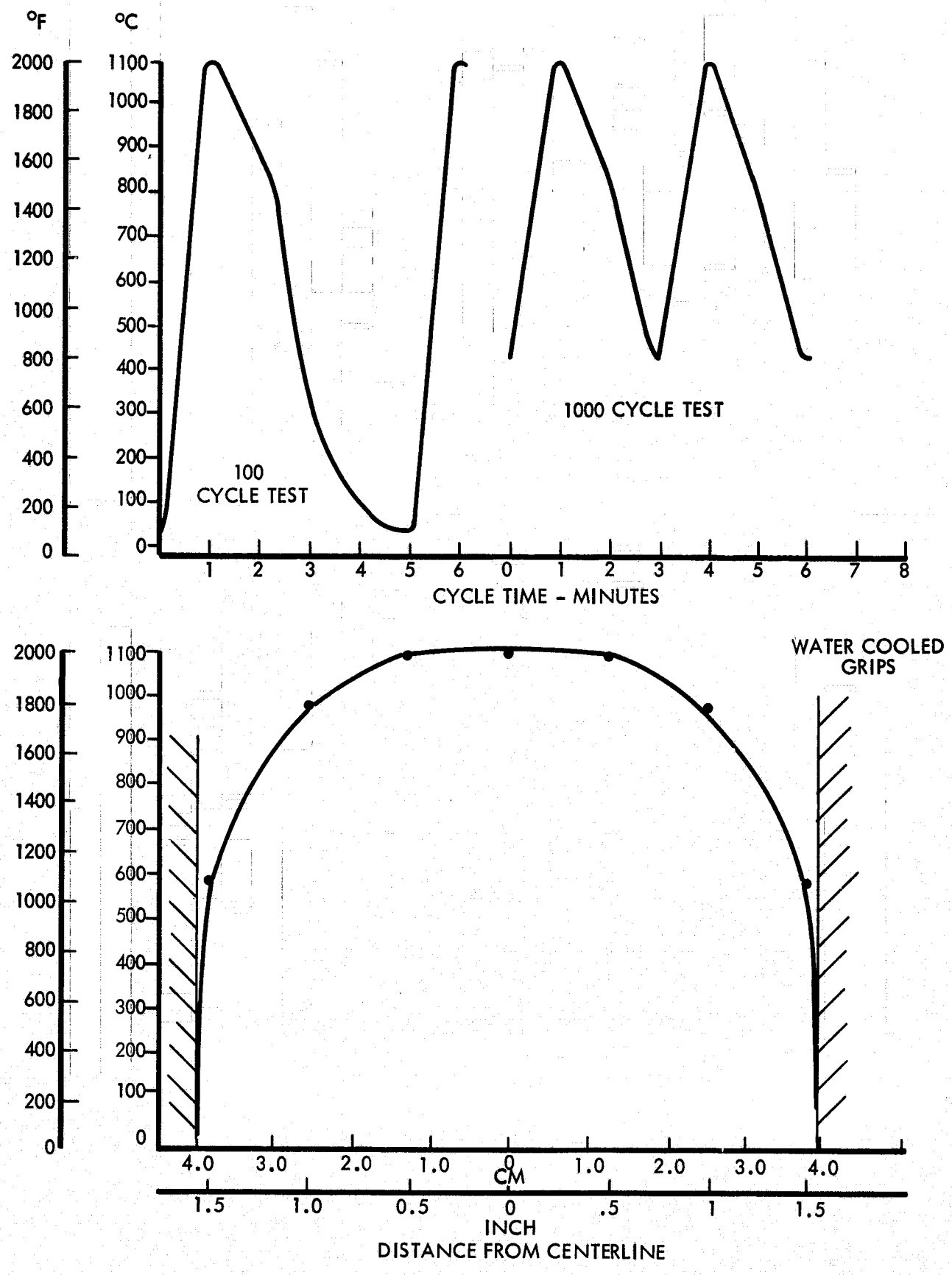


Figure 6. Thermal Cycles and Temperature Gradient Along Specimen During Thermal Fatigue Test.

about 2mm along the specimen. The length of specimen between grips was kept constant at 7.94 cm (3.125 inch). The total specimen length was about 13 cm (5 inch) so that approximately 2.5 cm (1 inch) was inside the cooled grips.

Platinum wires spaced about 1.3 cm (0.5 inch) apart were welded to the specimens to act as markers for optical measurement of axial strain during the test. Specimens were also evaluated for other dimensional changes, width, thickness and warpage and by metallographic cross sectioning at the end of the test.

Results of Thermal Fatigue Screening Studies

Matrix Alloy/Volume Fraction Relationship

The following systems were fabricated to investigate the effect of different strength matrix alloys and different volume fraction reinforcement levels on thermal fatigue behavior:

35v/o W-Ni/1
50v/o W-Ni/1

35v/o W-Ni/2
50v/o W-Ni/2

35v/o W-Ni/3
50v/o W-Ni/3

Specimens were subjected to 100 cycles between 38° and 1093°C and the dimensional changes noted. Specimens were then sectioned metallographically in the center of the gage section and examined for delaminations, fiber breaks, etc.

The measured dimensional changes are shown below. Changes in length refer to the total length of the specimen, not the 1/2 inch gage section which was used to monitor the test, but it was observed that at least 80% of the measured change occurred in the gage section.

	Specimen Dimensional Change					
	Thickness		Width		Length	
System	cm	inch	cm	inch	cm	inch
50v/o W-Ni/1	+.0018	+.0007	+.0038	+.0015	-.0014	-.0045
35v/o W-Ni/1	+.0022	+.0085	+.0261	+.0103	-.2857	-.1125
50v/o W-Ni/2	+.0030	+.0012	+.0038	+.0015	+.0005	+.0002
35v/o W-Ni/2	+.0120	+.0047	+.0068	+.0027	-.0660	-.0260
50v/o W-Ni/3	+.0096	+.0038	+.0068	+.0027	-.0416	-.0164
35v/o W-Ni/3	+.0564	+.0222	+.0561	+.0221	-.5862	-.2318

In all cases the dimensional changes decreased with increasing W reinforcement level as would be anticipated. The Ni/3 matrix (2-1DA) resulted in the greatest dimensional instability indicating highest matrix strength over the cycling temperature range which resulted in greater compressive deformation of the tungsten wires. A high thermal expansivity of this matrix alloy compared to the other alloys would also cause greater deformation of the wires. Comparative thermal expansivity data were not available however.

Metallographic evaluations were performed on the tested specimens. A measurable reaction zone was formed with the highest reaction rate indicated for the Ni/1 matrix. In the case of the specimens with greatest dimensional change, a measurable increase of fiber diameter was observed. No loss of fiber/matrix bond was observed even though compressive deformations of up to 15% in the tungsten fibers were observed. These data are shown below:

	Reaction Zone		Fiber Diameter Change	
System	μm	(inches) x 10^{-3}	cm	(inches)*
50v/o W-Ni/1	6.3	0.25	-	None
35v/o W-Ni/1	6.3	0.25	0.0028	+0.0011
50v/o W-Ni/2	3.0	0.12	-	None
35v/o W-Ni/2	2.5	0.10	-	None
50v/o W-Ni/3	2.5	0.10	-	None
35v/o W-Ni/3	2.5	0.10	0.0058	+0.0023

*Measured at the center.

Corresponding surface appearance and microstructures are shown in the following series of photographs (Figure 7). All of the specimens with 35v/o reinforcement level showed signs of warpage and/or surface delamination, the least amount of damage being exhibited by the NiCrAlY matrix system which is the most ductile and was the most fully densified matrix. No evidence of extensive internal matrix or fiber cracking was observed in any of these specimens. With the 50v/o reinforced specimens, there were no external or internal indications of thermal cycling damage.

Effect of Cross Plying

One system was selected to evaluate the response of a cross plied material to thermal cycling. The system selected by NASA was 35v/o W-Ni/2 in a balanced ply configuration of +15°, -15°, -15°, +15°. After 100 cycles between 39ª1093°C (100ª2000°F) under zero load conditions, the dimensional changes were as follows:

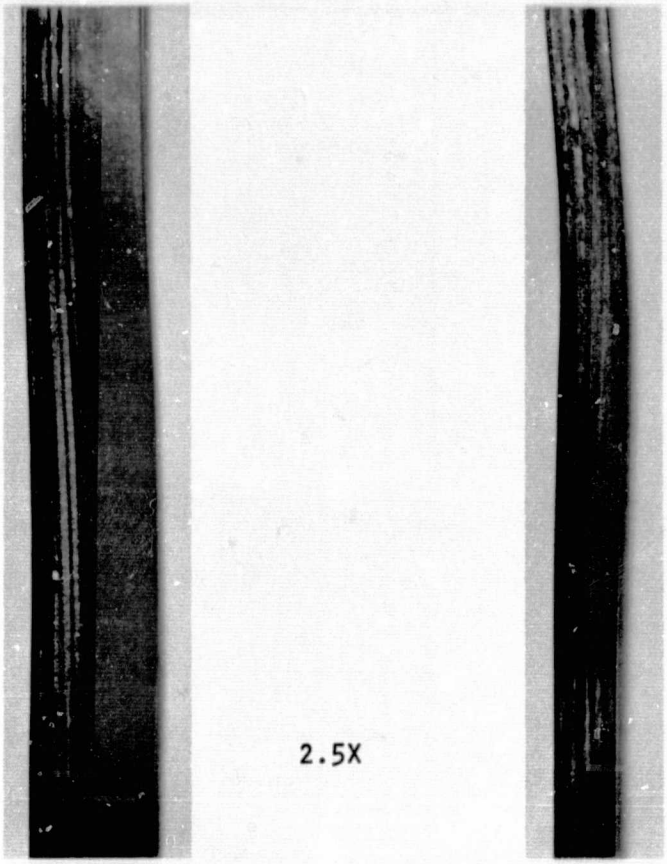
Thickness	Width	Length
+.0246 cm (+.0097 inch)	+.010 cm (+0040 inch)	-.226 cm (-.0891 inch)

The total shrinkage therefore being about 3-1/2 times greater than for a similar 0° uniaxial specimen. The measured increase in fiber diameter was much less than observed for the 35v/o W-Ni/1, 0° system in which the specimen dimensional changes were similar, showing that the resolved thermal stresses on the fibers were much lower in the cross-plied material. Additional mechanisms for stress relaxation now exist which are (1) fiber rotation and (2) matrix shear deformation. These effects could occur without disrupting fiber/matrix bonds and more extensive deformation, compared to specimens with 0° reinforcements, would be anticipated.

Visually the specimen showed evidence of surface microcracking, but no macroscopic distortion was evident. The microcracks did not appear to propagate into the sample cross section beyond about 0.76 mm (.0300 inch). Figure 8 shows the longitudinal (edge) section in the as-fabricated condition and transverse section (normal to specimen axis) after thermal cycling. No internal matrix or fiber cracking developed as a result of thermal cycling.

Matrix Alloy Condition

It was intended to compare specimens fabricated from matrix in the form of pre-alloyed powder and wrought sheet using the 2-1DA composition (Ni/3 and Ni/4 matrices). Problems were encountered in fabricating specimens with the sheet alloy at least in part due to the difficulty in preparing sufficiently clean metal surfaces, to allow good matrix/matrix bonding, and to the use of "non-optimized" fabrication parameters. It was therefore decided to use the NiCrAlY composition for these experiments using NASA-supplied sheet material, since prior work had indicated that the weaker, carbide free alloy was easier to bond.



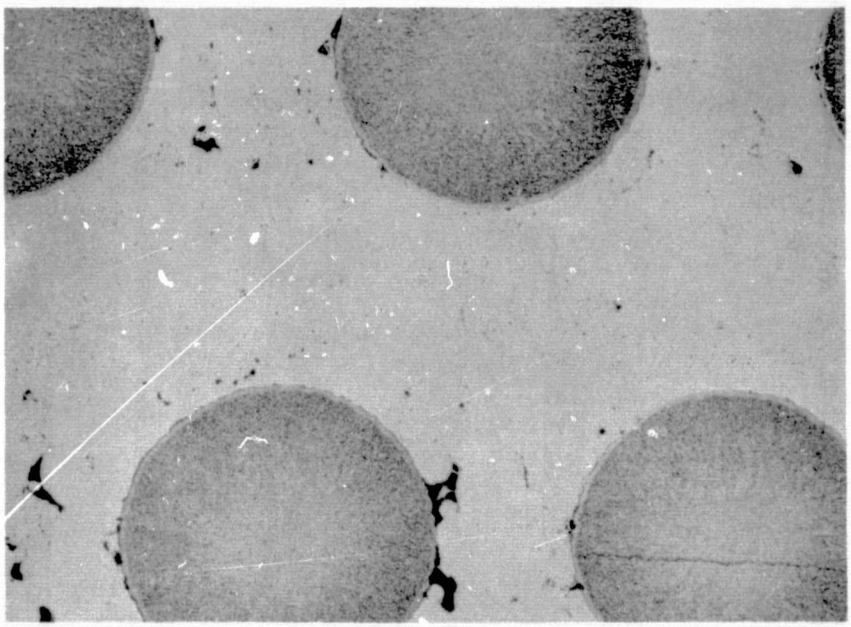
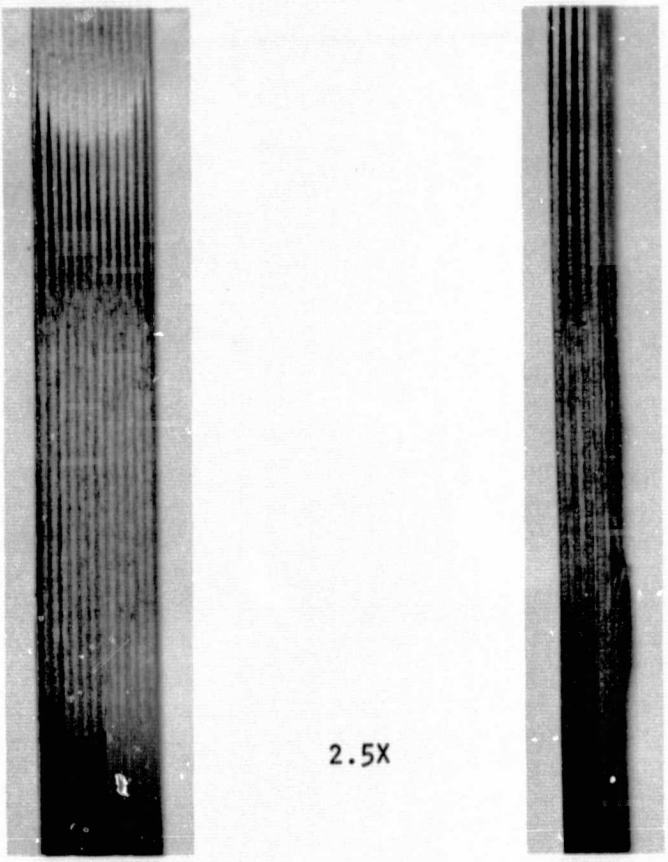


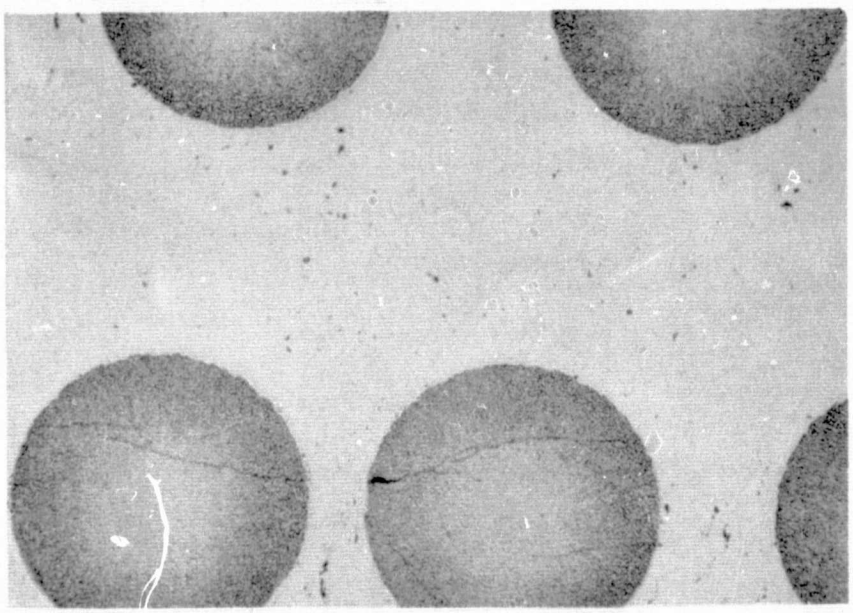
Figure 7. Macroscopic Appearance and Metallographic Cross Section of Thermal Fatigue Specimens After 100 Cycles Between 38 and 1093°C (100-2000°F).



2.5X

Front View

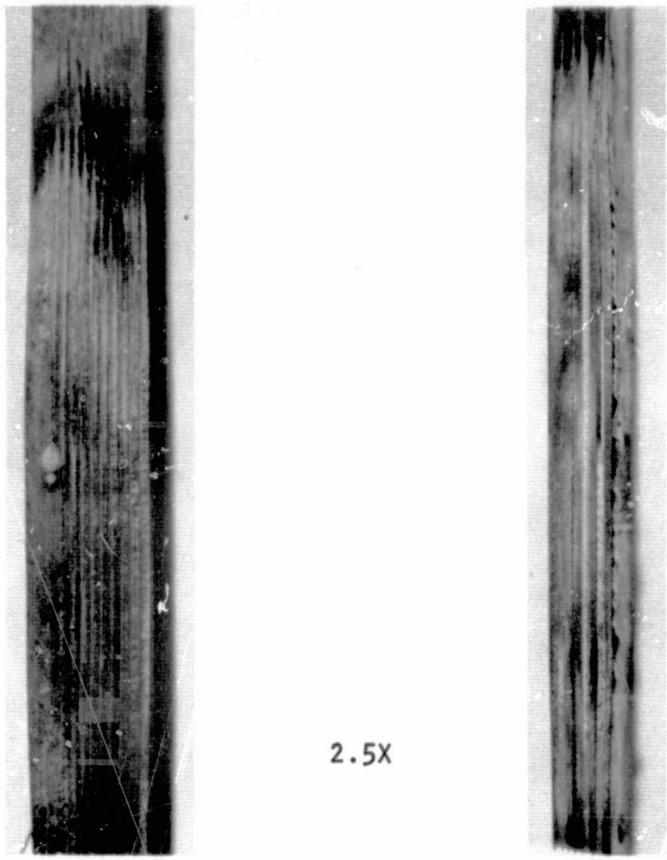
Side View



35v/o W-Ni/2

100X

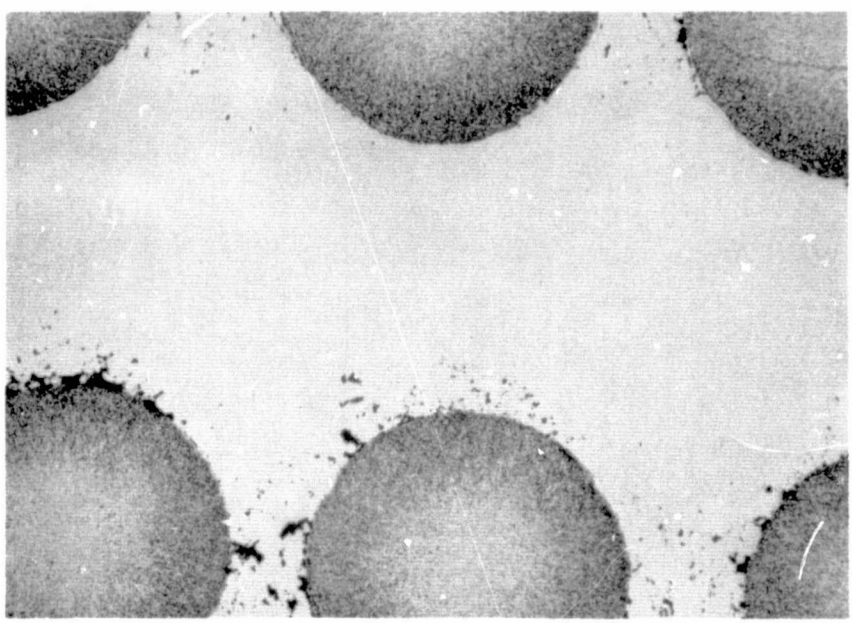
Figure 7 (contd.)



2.5X

Front View

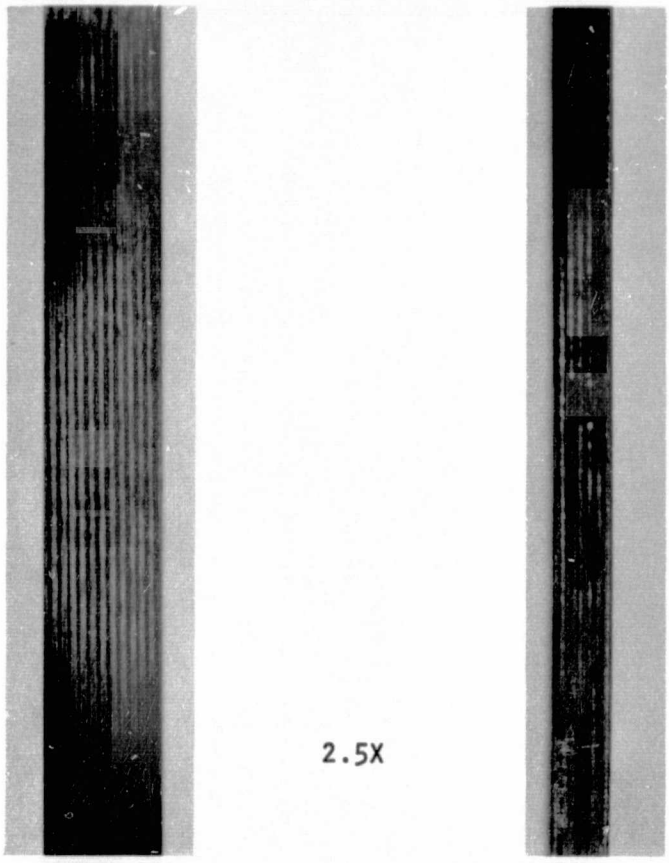
Side View



35v/o W-Ni/3

100X

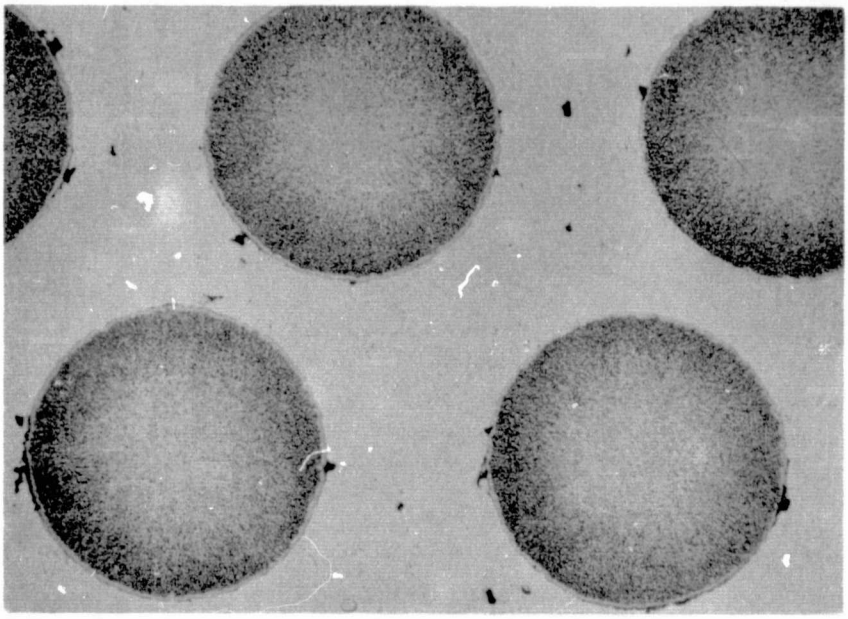
Figure 7. (contd)



2.5X

Front View

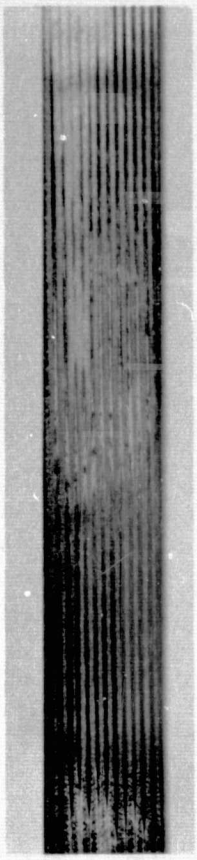
Side View



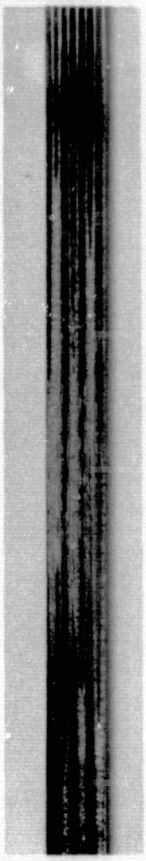
50v/o W-Ni/1

100X

Figure 7 (contd.)

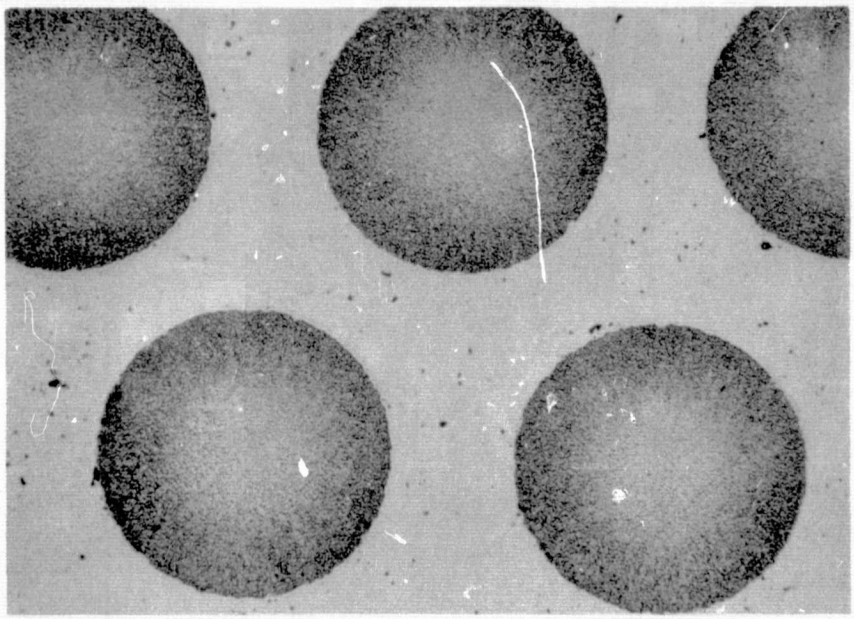


2.5X



Front View

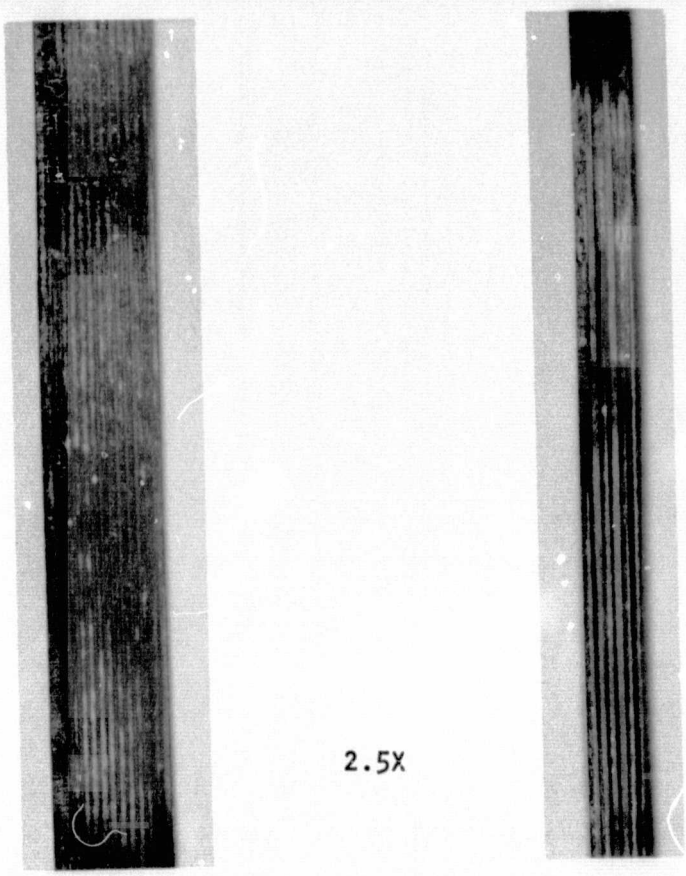
Side View



50v/o W-Ni/2

100X

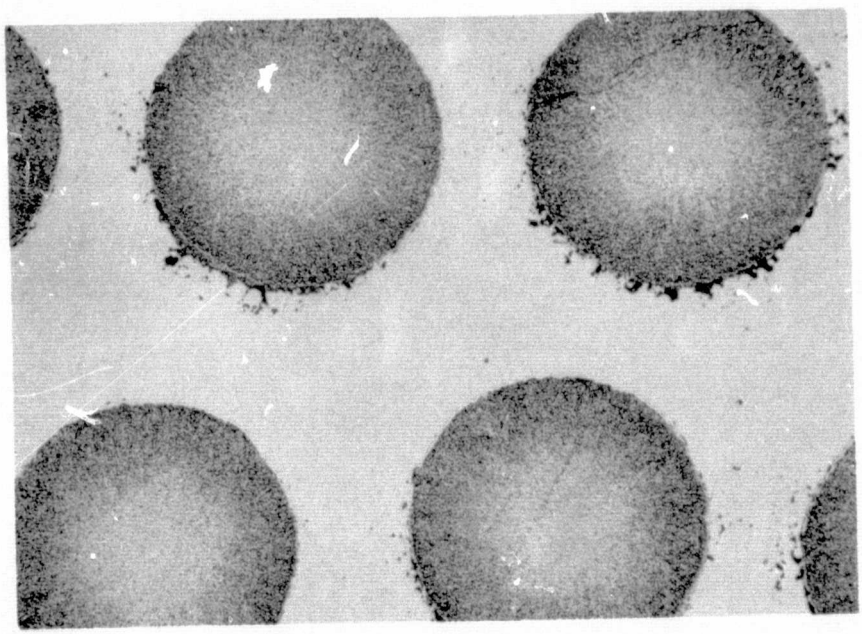
Figure 7 (contd.)



2.5X

Front View

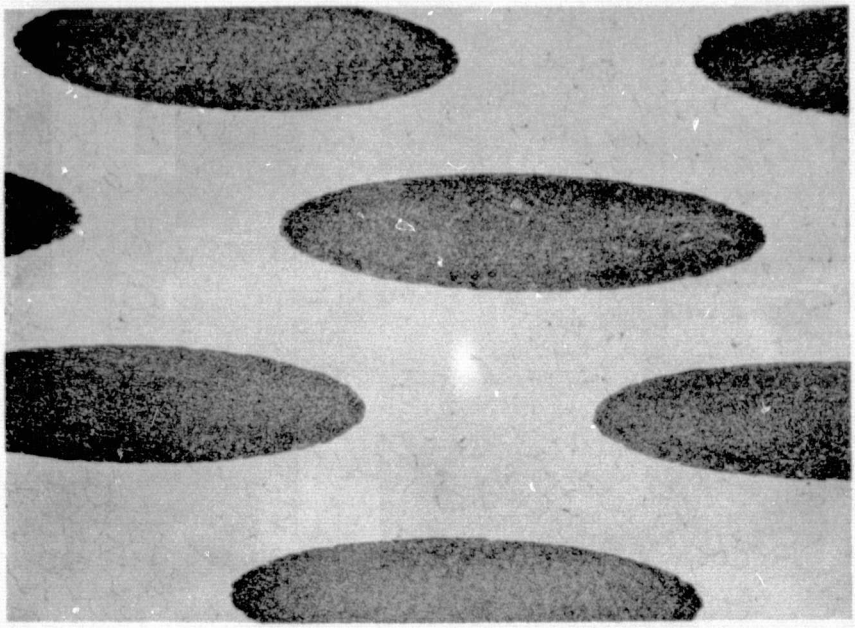
Side View



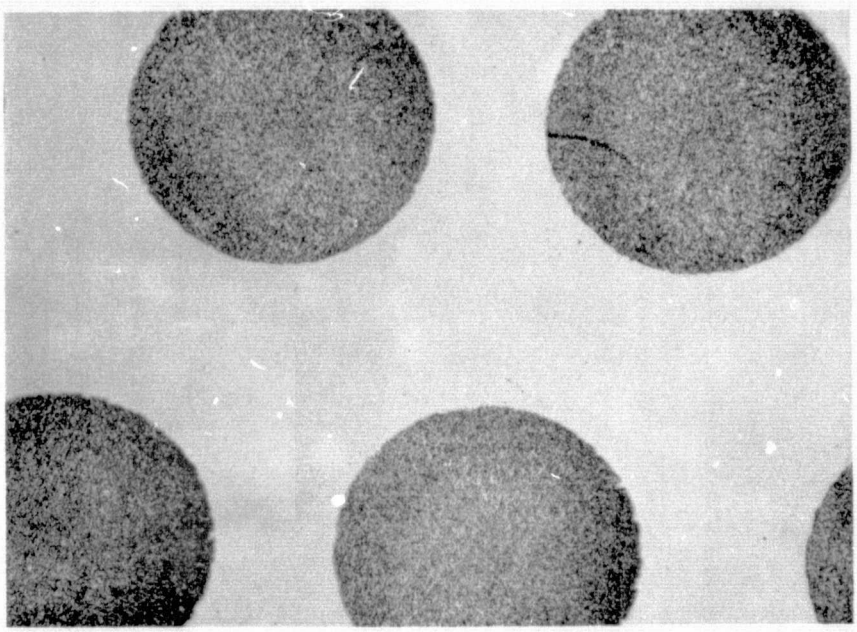
50v/o W-Ni/3

100X

Figure 7 (contd.)



A. Longitudinal (Edge) Section - As Fabricated. 40X



B. Cross Section - After 100 Cycles 100X

Figure 8. Cross Plied (±15°) 35v/o W-Ni/2 Specimen.

A. As Fabricated.
B. After 100 Cycles 38-1093°C (100-2000°F).

Panels were fabricated with 35v/o, 50v/o, and 65v/o reinforcements. The 65v/o specimen was shipped to NASA for evaluation. As shown in Figure 9, although the specimen panels appeared well consolidated, visually there was incomplete bonding at the matrix/matrix interface. The "as-fabricated" structures were again determined from the unaltered, water cooled gripping portion of tested samples, so that the problem of incomplete bonding was not established until the tests had been completed. The dimensional change data following 100 cycles of 38°-1093°C (100°-2000°F) are shown below compared with the previous data for the powder matrix material.

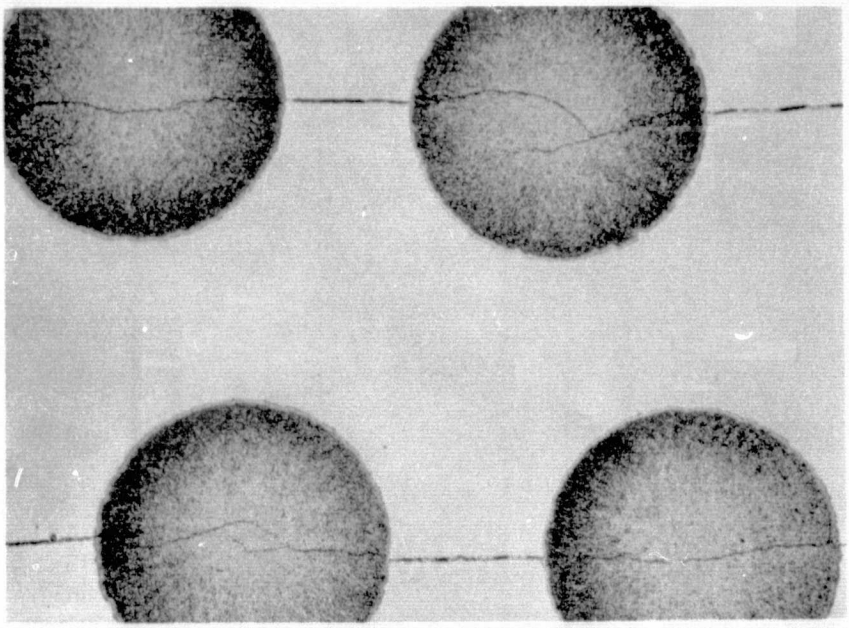
	Dimensional Changes					
	Thickness		Width		Length	
Matrix	cm	inch	cm	inch	cm	inch
50v/o W-Ni/5	-	-	+.0076	.0030	-.0101	-.0040
35v/o W-Ni/5	+.0002	+.0808	+.0043	+.0017	-.0272	-.0107
50v/o W-Ni/2	+.0030	+.0012	+.0038	+.0015	+.0005	+.0002
35v/o W-Ni/2	+.0120	+.0047	+.0068	+.0027	-.0660	-.0260

There was an apparent anomaly with the length change data since at the higher volume fraction level the sheet alloy matrix behaved as if it were a stronger alloy than the powder matrix while at the lower volume fraction level, the converse was true. Reference to the thickness measurement however shows that there was a disproportionate increase in thickness for the sheel alloy system. The data may be further rationalized by reference to the microstructures in Figures 9 and 10. At the 35v/o reinforcement level, matrix relaxation, due to the incomplete bonding, created sufficiently high transverse stresses in the fibers to cause cracking on the first cooldown cycle represented by the fabrication cycle. As a result of subsequent thermal cycles, some crack broadening occurred as the specimen swelled. This would provide a mechanism,other than ratchetting, of accommodating the thermally induced stresses. In the specimen with 50% reinforcement, stresses on the fibers were insufficient to cause transverse failure, even after thermal cycling (Figure 10) and a small amount (0.1% over the total specimen length) of negative creep was observed.

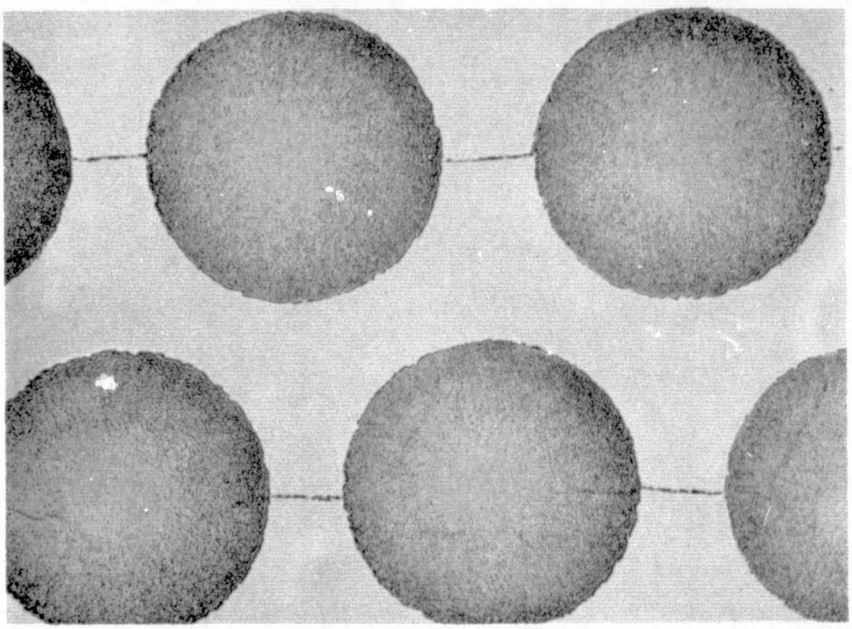
External Load Effects

The 35v/o W-Ni/2 system was selected by NASA to evaluate the effect of applying a static tensile load during testing on thermal fatigue behavior. A stress of 6 ksi was selected for application during thermal cycling. Dimensional changes following the standard 100 cycles (38°-1093°C) test were as follows:

Width	0
Thickness	+.0084 cm (.0033 inch)
Length	0,

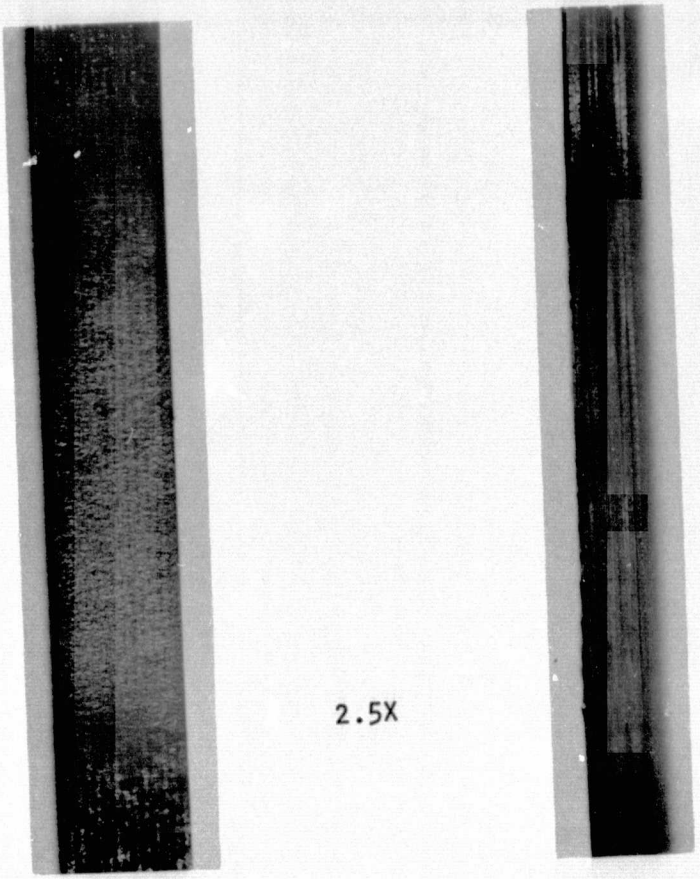


25v/o W-Ni/5 100X



50v/o W-Ni/5 100X

Figure 9. Cross Sections Through As-Fabricated W-Ni/5 Thermal Fatigue Specimens.



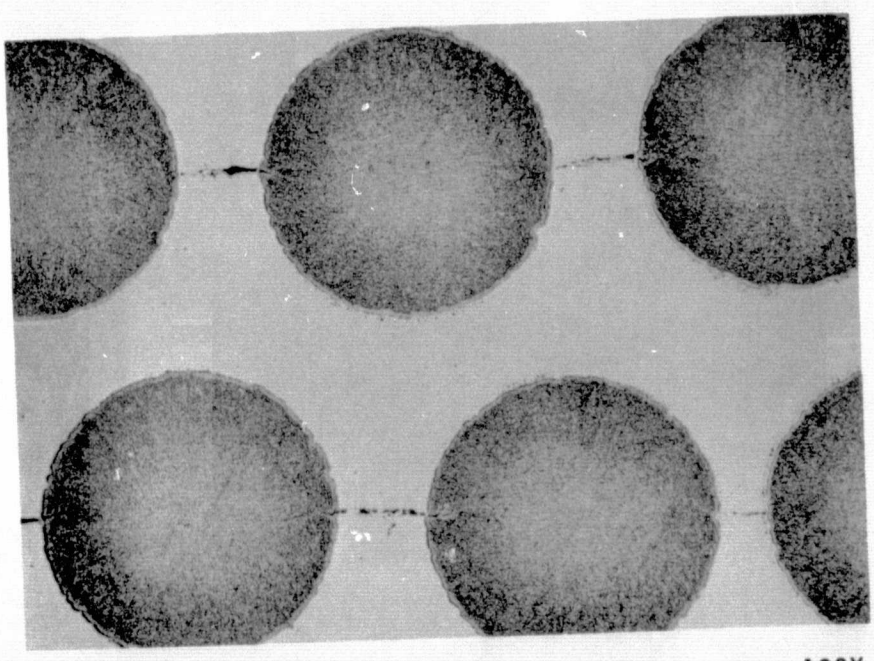


Figure 10. Macroscopic Appearance and Metallographic Cross Section of W-Ni/5 Thermal Fatigue Specimen.

i.e., the overall dimensional changes were considerably less than in the corresponding 0 load condition. There was no microscopically observable distortion or cracking after this test and metallographic cross sectioning did not reveal any internal damage or change in fiber diameter. The measured thickness increase in the gage section (amounting to about 6%) was apparently all due to plastic deformation in the matrix.

Matrix Conditioning

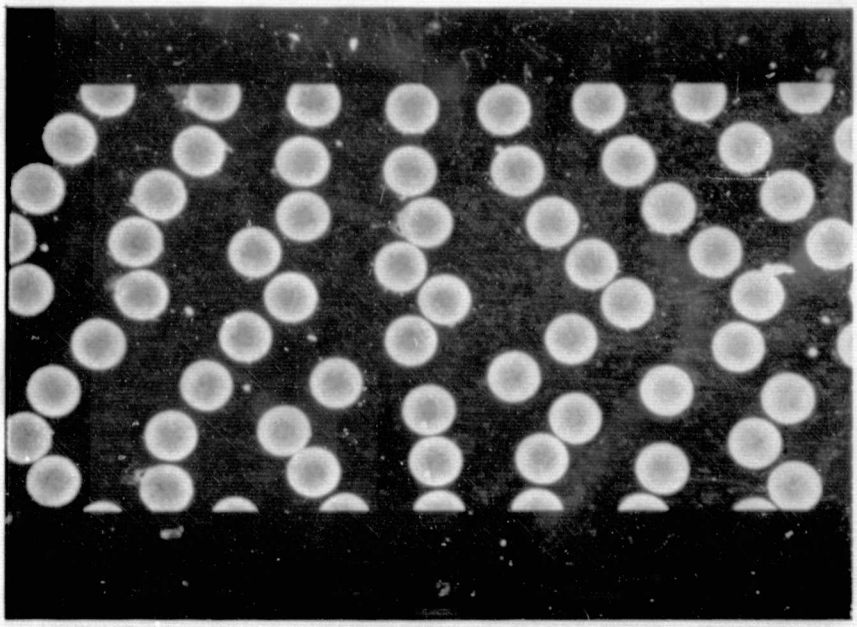
The 35v/o W-Ni/1 (NASA Alloy III Matrix) system was selected by NASA to investigate the effect of mechanical working on composite thermal fatigue behavior. The specimen was upset forged to 38% reduction and subjected to the standard 100 cycle 38°-1093°C (100-2000°F) test. Forging was selected as a means of mechanically working the matrix because of the more controlled conditions (compared to rolling or extrusion) although it was recognized from work at TRW[6] and elsewhere[7] that potentially undesirable filament redistributions could occur during large percentage forging reductions. In the case of ductile matrix/ductile filament composites, longitudinal rolling may represent a more attractive deformation processing method based on preliminary investigations.

Metallography showed that all of the microporosity present in the as-fabricated condition (see Figure 11.B) was removed. The macrophotograph in Figure 11.A shows the entire cross section of the machined thermal fatigue specimen and illustrates the filament redistribution into a "flow line" pattern. The fibers have moved closer together in the pressing direction and have spread outwards, in the horizontal plane, acting as an effective grid pattern for the deformation.

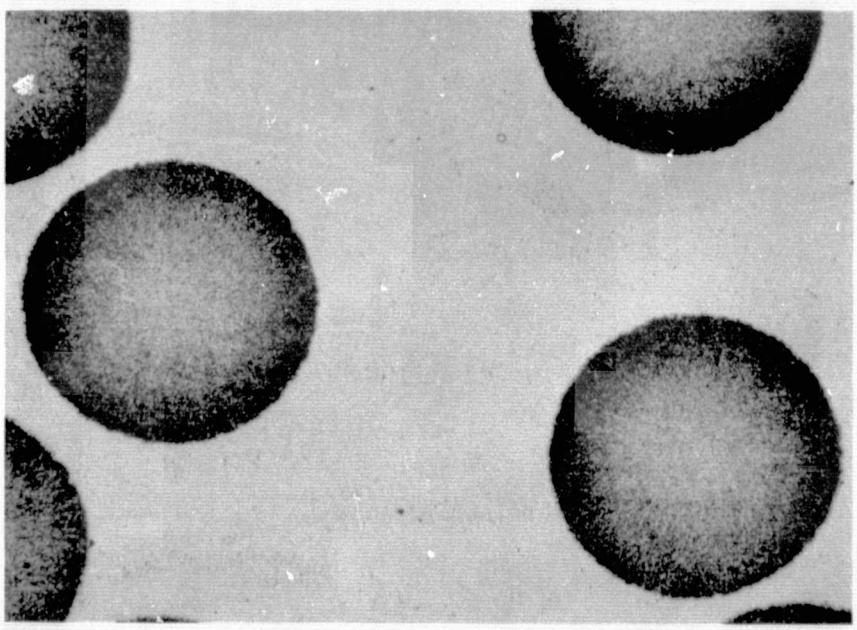
The measured dimensional changes following the 100 thermal cycles are tabulated below and compared to the equivalent 35 and 50v/o specimens in the as-fabricated condition.

	Specimen Dimensional Change					
	Thickness		Width		Length	
System	cm	inch	cm	inch	cm	inch
50v/o W-Ni/1	+.0018	+.0007	+.0038	+.0015	-.0014	-.0045
35v/o W-Ni/1	+.0022	+.0085	+.0261	+.0103	-.2857	-.1125
35v/o W-Ni/1 (forged)	+.0112	+.0044	+.0206	+.0081	-.1473	-.0580

There was a significant decrease (48%) in the amount of shrinkage after forging compared to the as-fabricated condition. The dimensional instability of the forged 35v/o specimen was still considerably greater than the as-fabricated 50v/o specimen, however, Figure 11.b Shows a metallographic cross section through the gage section of the tested specimen. No signs of cracks were visible in the matrix or fibers and additionally, the matrix appeared fully densified with no traces of powder particle boundaries.



A. Cross Section of Forged Specimen Showing Filement Redistribution. 18X



B. Gage Section of Specimen After 100 Cycles 38°-1093°C (100-2000°F). 100X

Figure 11. Cross Section of 35v/o W-Ni/1 (Reduction Forged) Thermal Fatigue Specimen.

Conclusions from the Thermal Fatigue Screening Studies

Major conclusions from these preliminary evaluations were as follows:

1. Specimen dimensional instability was a function of both fiber volume fraction and matrix strength and ductility.

2. Investigation on the effect of matrix alloy composition were complicated by the lack of densification and poor bonding in some cases, and the need for more extensive process optimization studies was identified.

3. The dimensional instability of an all cross-ply ($\pm 15°$) system, tested with no applied load, was significantly greater than for the corresponding unidirectionally reinforced system.

4. Application of a small axial load (equivalent to 6 ksi) during thermal cycling of a 35v/o reinforced composite, largely eliminated dimensional changes.

5. Mechanically working the matrix by a secondary fabrication process (hot forging) was shown to be effective in promoting full densification and in improving dimensional stability under thermal cycling conditions. The reasons for reduced axial shrinkage, compared to a specimen in the as-fabricated condition and containing voids, were not clear. Possibly this was due to the development of a more ductile matrix.

6. The most ductile (and easily fabricated) NiCrAlY matrix system showed the greatest potential of meeting the immediate program goals - namely to successfully withstand 1000 thermal cycles, 427°-1093°C (800°-2000°F) without sustaining significant damage.

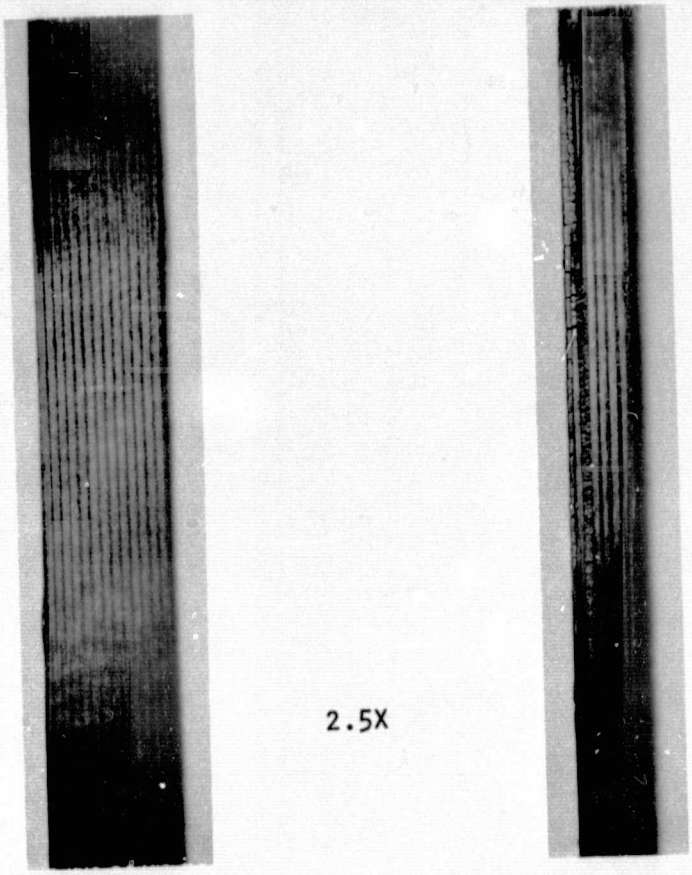
Advanced Thermal Fatigue Characterization Studies

For these investigations, the thermal cycles were 427°-1093°C (800° - 2000°F) with a target of 1000 cycles. Specimens were run with 0, 6 and 12 ksi applied stress. The dimensional change data are summarized below:

System	No. of Cycles	Applied Tensile Stress (ksi)	Specimen Dimensional Change					
			Thickness		Width		Length	
			cm	inch	cm	inch	cm	inch
50v/o W-Ni/2	1000	0	+.0205	+.0081	+.0096	+.0038	-.0010	+.0004
35v/o W-Ni/2	1000	6	+.0172	+.0068	-.0033	-.0013	-.0746	-.0294
35v/o W-Ni/2	876	12	Failed in Test.					
35v/o W-Ni/5*	1000	6	+.0434	+.0171	+.0122	+.0048	+.0058	+.0023

* NiCrAlY Sheet

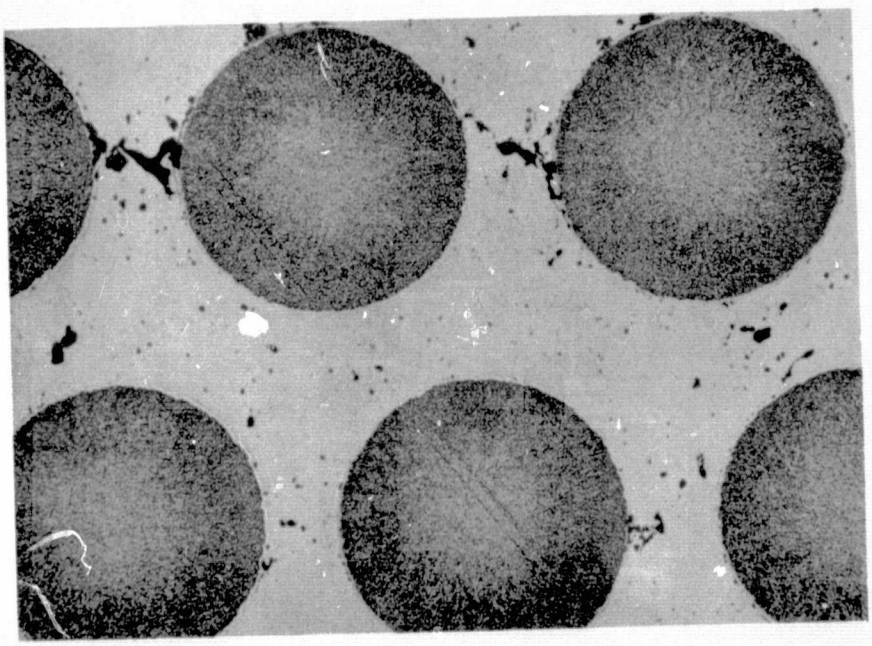
The 50v/o W-Ni/2 system showed very little dimensional change after 1000 cycles with zero applied load. The 35v/o W-Ni/2 after 1000 cycles with a 6 ksi applied tensile stress showed similar dimensional changes to the same system under zero applied stress after 100 cycles. Dimensional changes in the 35v/o W-Ni/5 system were all positive. This was explained by metallographic cross sectioning which showed that considerable delamination occurred at the foil/foil interfaces. Metallogrpahy also indicated that the specimens fabricated from the powder matrix material had marginal bonding between the larger powder particles and internal microcracking occurred. Figure 12 shows the appearance of the 50v/o W-Ni/2 specimen after test from which surface microcracking on the edge and internal cracks or voids which developed, mostly between fibers can be seen. These effects were more severe in the case of the 35v/o reinforcement level as indicated in Figure 13. A problem of localized overheating may develop due to void formation, since changes in resistivity and conductivity occur and the control thermocouple at the surface may not detect internal overheating. An indication of this effect is the amount of recrystallization which takes place in the tungsten wire due to the localized overheating. An example of this effect is shown in Figure 14 for the 35v/o W-Ni/2 specimen loaded to 6 ksi. One row of fibers showed recrystallization induced by fairly extensive fiber/matrix interaction, whereas the other fibers showed negligible reaction characteristic of this thermal exposure. In the extreme case, matrix melting can occur, with subsequent failure. Overheating effects would probably explain the premature fialure (876 cycles) of the loaded specimen tabulated above. Based on these data, it was decided to fabricate specimens for Task II evaluations using higher temperature and/or pressure parameters to ensure full densification and adequate metallic bonding.



2.5X

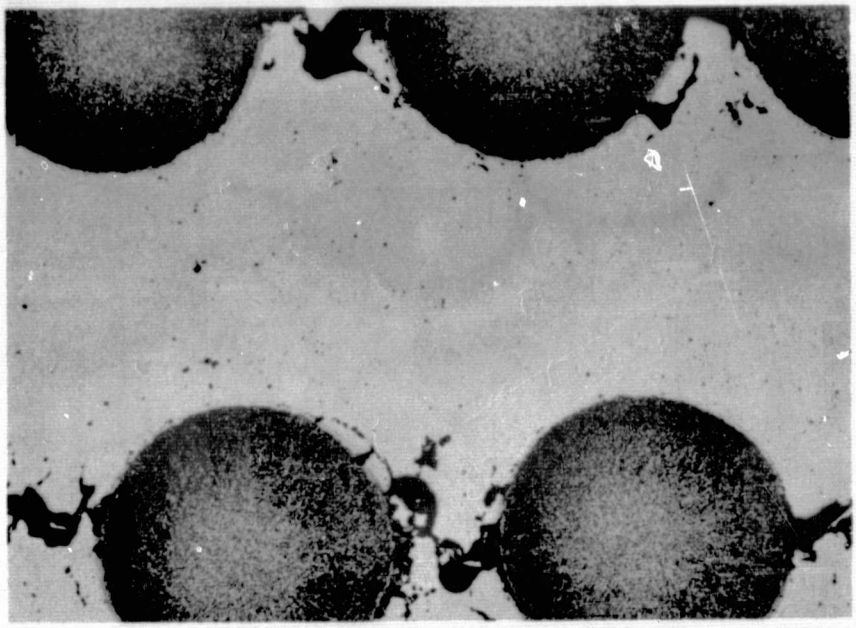
Front View

Side View



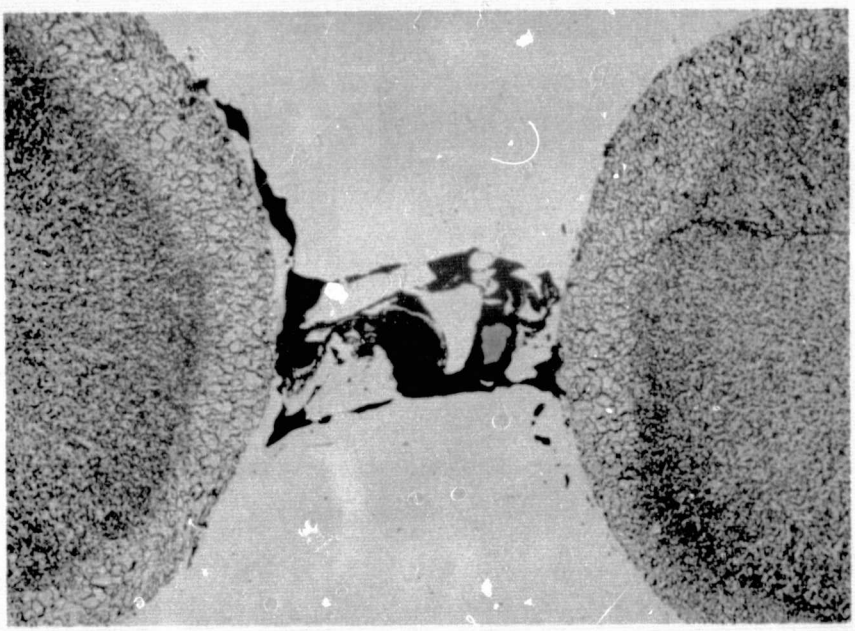
100X

Figure 12. Surface Appearance and Metallographic Cross Section of 50v/o W-Ni/2 Thermal Fatigue Specimen After 1000 Cycles 426-1093°C (800-2000°F).



100X

Figure 13. Cross Section Through 35v/o W-Ni/2 Specimen After 1000 Cycles.



Note Recrystallized Fibers 250X

Figure 14. Evidence of Overheating in 35v/o W-Ni/2 Specimen After 1000 Cycles 426-1093°C (800-2000°F). Static Load Applied-6 KSI.

Composite Characterization Studies

Based on the results of the thermal fatigue evaluations, two matrix alloy systems were selected for additional evaluations which included 1093°C (2000°F) stress rupture, impact tests and static oxidation.

Systems Selection

The composite systems selected for evaluation in Task II by the NASA technical program director were as follows:

Stress Rupture	-	50v/o W-Ni/2 35v/o W-Hf-C-Ni/2
Impact	-	50v/o W-Ni/2 50v/o W-Ni/1
Oxidation	-	50v/o W-Ni/2 50v/o W-Ni/1

All specimens, except for the impact test specimens, were to have completely protected fibers with a target minimum of .0508 cm (.020 inch) of protective cladding.

Stress Rupture

Stress rupture evaluations were carried out on 50v/o W-Ni/2 and 35v/o (W-Hf-C)-Ni/2 systems. The high strength fiber material was supplied by NASA.

Specimen Fabrication

There was some concern initially that the advanced W-Hf-C filament might prove difficult to collimate by standard drumwinding processes. No problems were in fact encountered.

A two-stage fabrication process was used to produce panels from which completely clad stress rupture specimens could be machined. Panels with the required number of plies were fabricated from which parallel sided strips were machined for subsequent cladding, using the same matrix composition. The panels were designed to have 50 and 65v/o packing densities to subsequently provide specimens with nominal overall reinforcement levels of about 35 and 50 volume fraction. Stress rupture specimens of the configuration shown in Figure 15 (without the reduced gage width) were machined by EDM techniques.

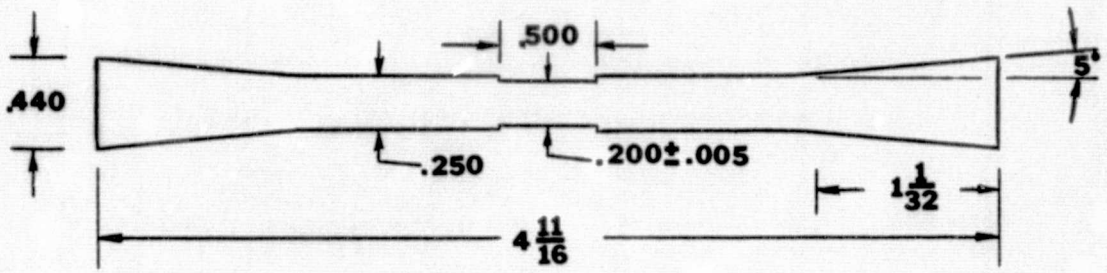


Figure 15. Stress Rupture Test Specimen Design.

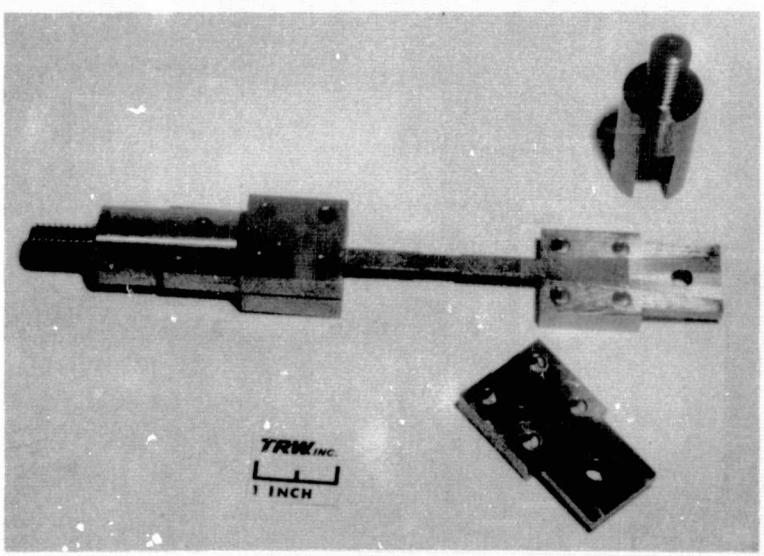


Figure 16. Stress Rupture Grip Assembly and Machined Specimen.

The B-1900 nickel superalloy grip fixture which was used is shown in Figure 16. Tests were conducted in a static air environment and the specimens had thermocouples in tight contact with the gage section to monitor temperature.

The data from these tests are shown below in Table II and again in a stress vs. log time plot in Figure 17. A semi-log least squares linear regression analysis was performed to fit the data points in Figure 17. The indicated stress levels for 100-hour rupture are about 179 MN/m^2 (26.5 ksi) and 365 MN/m^2 (53 ksi) for W-Ni/2 and W-Hf-C-Ni/2 systems respectively. Average overall reinforcement levels were about 40 and 30 volume percent so that very significant 100-hour rupture strength advantage (on a specific strength basis) was demonstrated for W-Hf-C/NiCrAlY. The actual volume fraction reinforcement levels of representative specimens were determined by metallographic cross sectioning, after test. This was achieved by determining the total fiber area, including partly machined fibers in the core but not including fibers that were visibly exposed on the specimens after test. Cross sections showing examples of good fiber distribution and exposed fibers due to machining which have subsequently been lost by oxidation are shown in Figures 18 and 19. The specimen in Figure 18 had a relatively well distributed core with a slight excess of matrix material on the edges. This specimen was step loaded and received a total 1093°C 2000°F exposure time of 248 hours. During machining of Specimen 104B, 3 fibers were exposed at one edge and were subsequently oxidized during tests. As indicated in Figure 19, the neighboring fibers were unaffected. Figure 20 shows typical cross sections of these W-Hf-C/NiCrAlY specimens at higher magnification and identified fiber/matrix reaction zone thicknesses. The measured reaction zone thicknesses are consistent with the data reported by Brentnall et al[8] for W-FeCrAlYcomposites.

Miniature Izod Impact Tests

Notched impact tests were performed following the methods of Winsa and Petrasek[3]. Tests were performed at room temperature, 149°C (300°F), 260°C (500°F), and 371°C (700°F) on 50v/o 218 W-Ni/1 and 50v/o 218 W-Ni/2 composite specimens. In the elevated temperature test, specimens were heated by a hot air blower or propane torches and temperature was monitored continuously using an X-Y recorder and a thermocouple which was tack welded to the specimen within 1-2 mm of the notch. The specimens were heated to approximately 29C° (50F°) higher than the required temperature and the specimens were impact loaded on the cool-down cycle. Using this technique, the temperature at the moment of impact was usually within 3C° (5F°) of the required test temperature. The appearance of specimens after test is shown in Figure 21.

Data from these tests are summarized in Table III and shows that these materials have a definite ductile-brittle transition range. These values are somewhat higher than those reported by Winsa and Petrasek on notched (and unnotched at RT and 300°F) as-HIP tungsten/superalloy composites at similar reinforcement levels. The significant increases in RT impact strengths which were reported after mechanical working would also be expected to accrue in these current systems.

TABLE II

1093°C (2000°F) Air Environment Stress Rupture Data

Specimen	System	Applied Stress MN/m²	Applied Stress ksi	Time To Rupture Hours	Actual Volume Fraction
95 C	50v/o W-Ni/2	220	32	0.6	41
96 C	"	220	32	1.2	ND
96 A	"	207	30	3.3 Exposed Fiber	ND
95 B	"	207	30	21.3	42
96 D	"	158	23	115.6+	38
"	"	172	25	126.6+	"
"	"	196	28.5	65.3	"
107 B	35v/o(W-Hf-C)-Ni/2	448	65	Failed In Grip	ND
105 B	"	448	65	0.9 Exposed Fiber	29
106 B	"	413	60	13.1	ND
104 B	"	413	60	31.3	31*
105 A	"	344	50	139.6	ND
106 A	"	324	47	166.1+	28
"	"	344	50	82.1	

* Edge Exposed Fibers Not Included.

ND- Not Determined.

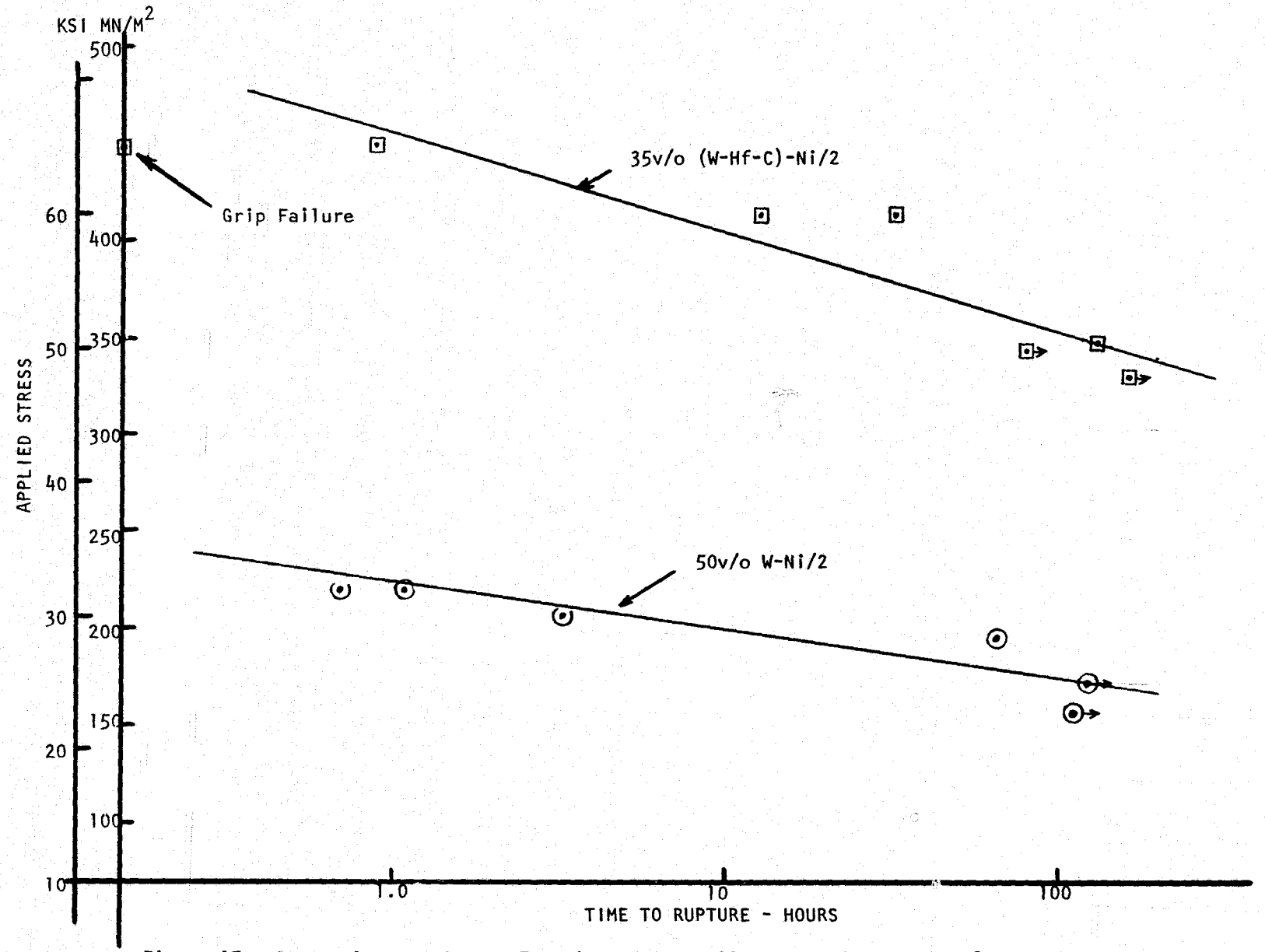
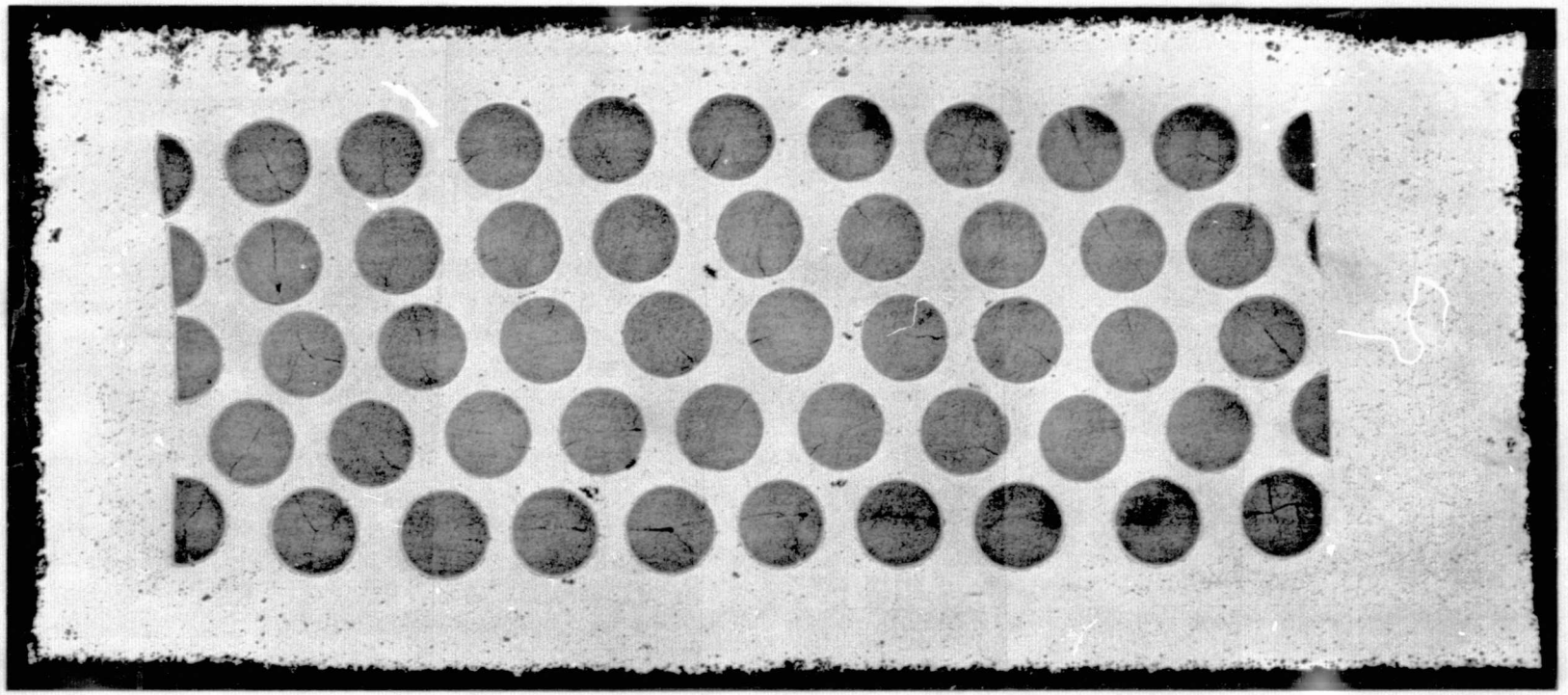
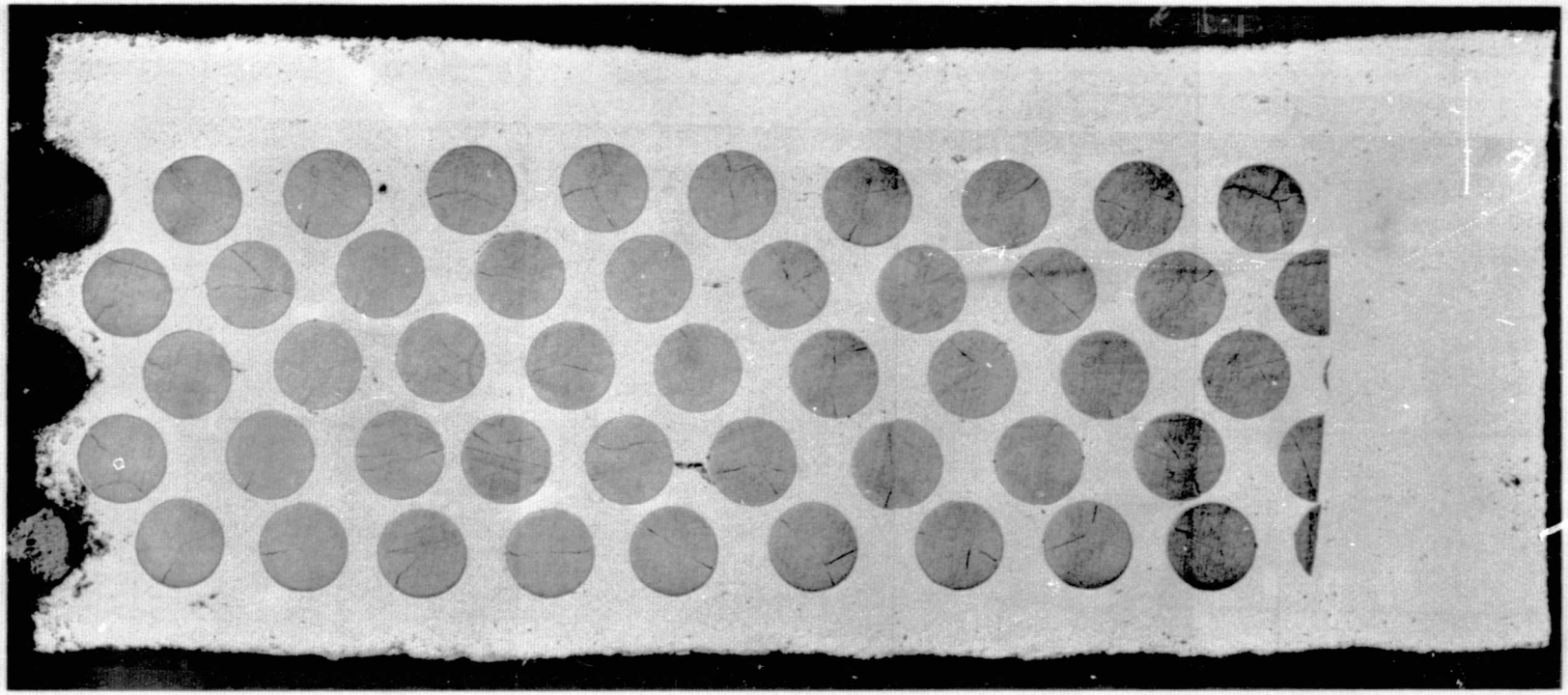


Figure 17. Stress Rupture Curves For Ni-Base Superalloy Composites with 218CS and W-Hf-C Fiber Reinforcements



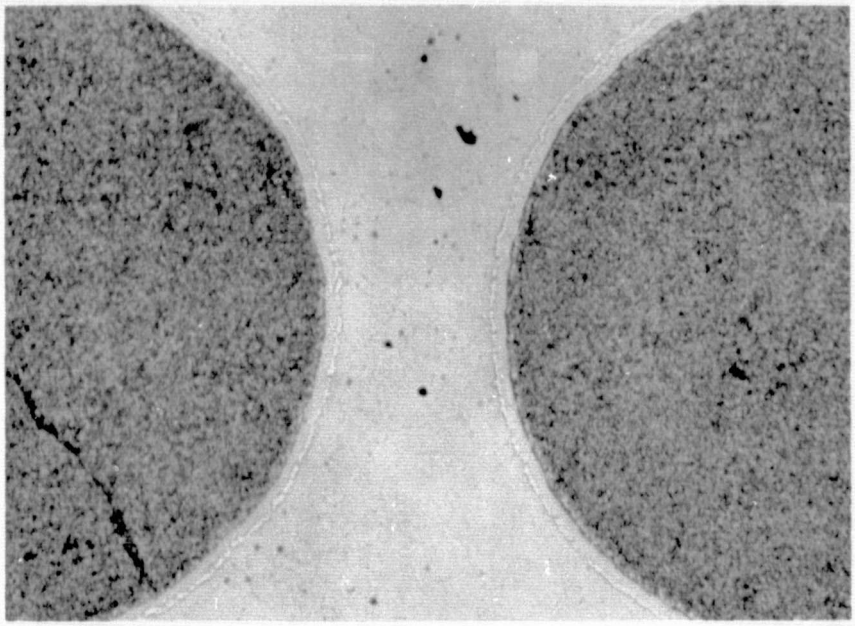
35X

Figure 18. Metallographic Section Adjacent to Fracture Zone in 35v/o (W-Hf-C)-Ni/2 Stress Rupture Specimen.
Overall Volume Fraction = 0.28. Specimen Exposure - 2000°F, 47 KSI, 166 Hours
+2000°F, 50 KSI, 82 Hours.



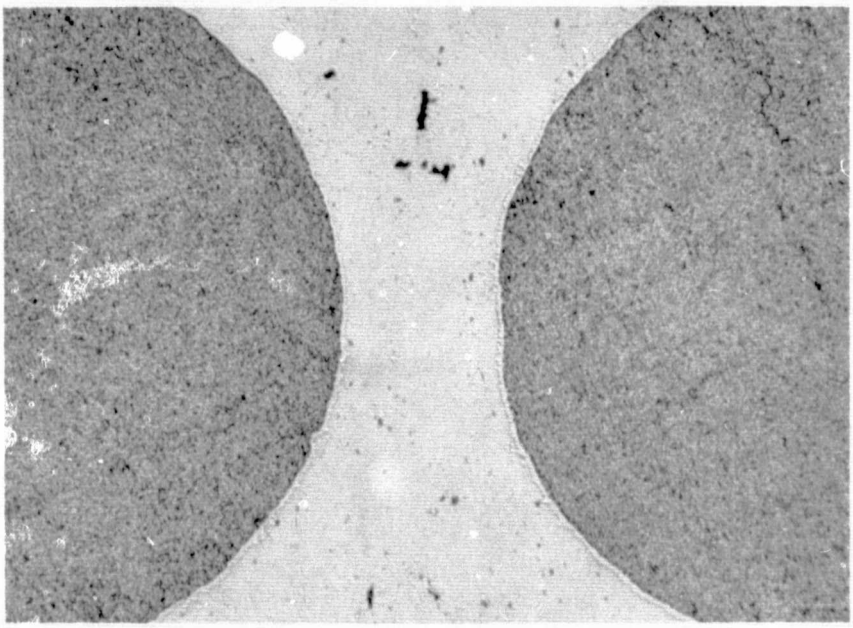
Note Fiber Loss Due to Machined-Through Fibers 35X

Figure 19. Metallographic Section Adjacent to Fracture Zone in 35v/o (W-Hf-C)-Ni/2 Stress Rupture Specimen. Overall Volume Fraction = 0.31 Specimen Exposure - 2000°F, 60 KSI, 31.3 Hours.



250X

2000°F - 248 Hours.Reaction Zone = 8.8 µm.



250X

2000°F 31.3 Hours. Reaction Zone = 4 µm.

Figure 20. Fiber/Matrix Reaction Zone Thicknesses in Stress Rupture Specimen.

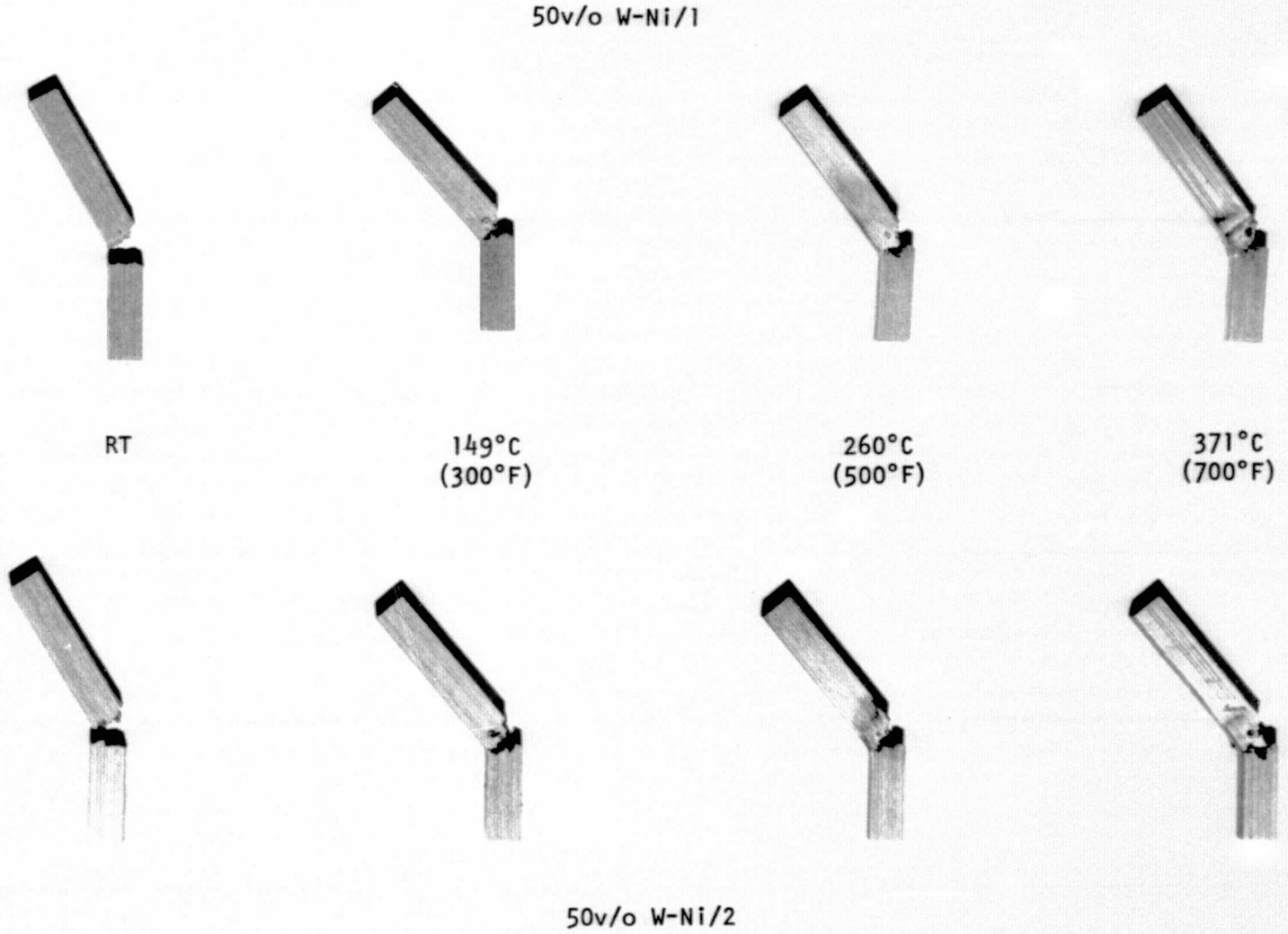


Figure 21. Appearance of W-Superalloy Miniature Izod Impact Specimens After Test.

TABLE III

Notched Impact Strengths of W-Superalloy Composites

Spec. I.D.	System	Test Temperature °F	Test Temperature °C	Joules	In-Lb	Normalized Impact Energy In-Lb/In^{2*}
102-1	50v/o W-Ni/1	75	23.8	.84	7.5	241.9
102-2	"	"	"	.62	5.5	177.4
102-3	"	300	148.9	1.30	11.5	370.9
102-4	"	"	"	1.36	12.0	387.1
102-5	"	500	260.0	4.35	38.5	1241.9
102-6	"	"	"	4.74	42.0	1354.8
101-7	"	700	371.0	4.57	40.5	1306.4
101-8	"	"	"	4.24	37.5	1209.7
81-1	50v/o W-Ni/2	75	238.0	.79	7.0	225.8
81-2	"	"	"	.73	6.5	209.6
80-1	"	300	148.9	1.36	12.0	387.1
80-2	"	"	"	.96	8.5	276.2
82-1	"	500	260.0	2.54	22.5	725.8
82-2	"	"	"	3.50	31.0	1000.0
78-1	"	700	371.0	10.73	95.0	3064.5
78-2	"	"	"	5.42	48.0	1548.4

* Based on Average Specimen Area of 0.200 cm^2 (0.031 in^2).

1 J = 8.85 inch lb = .737 ft. lb.

TABLE III

Notched Impact Strengths of W-Superalloy Composites

Spec. I.D.	System	Test Temperature °F	Test Temperature °C	Joules	In-Lb	Normalized Impact Energy In-Lb/In^{2*}
102-1	50v/o W-Ni/1	75	23.8	.84	7.5	241.9
102-2	"	"	"	.62	5.5	177.4
102-3	"	300	148.9	1.30	11.5	370.9
102-4	"	"	"	1.36	12.0	387.1
102-5	"	500	260.0	4.35	38.5	1241.9
102-6	"	"	"	4.74	42.0	1354.8
101-7	"	700	371.0	4.57	40.5	1306.4
101-8	"	"	"	4.24	37.5	1209.7
81-1	50v/o W-Ni/2	75	238.0	.79	7.0	225.8
81-2	"	"	"	.73	6.5	209.6
80-1	"	300	148.9	1.36	12.0	387.1
80-2	"	"	"	.96	8.5	276.2
82-1	"	500	260.0	2.54	22.5	725.8
82-2	"	"	"	3.50	31.0	1000.0
78-1	"	700	371.0	10.73	95.0	3064.5
78-2	"	"	"	5.42	48.0	1548.4

* Based on Average Specimen Area of 0.200 cm^2 (0.031 in^2).

1 J = 8.85 inch lb = .737 ft. lb.

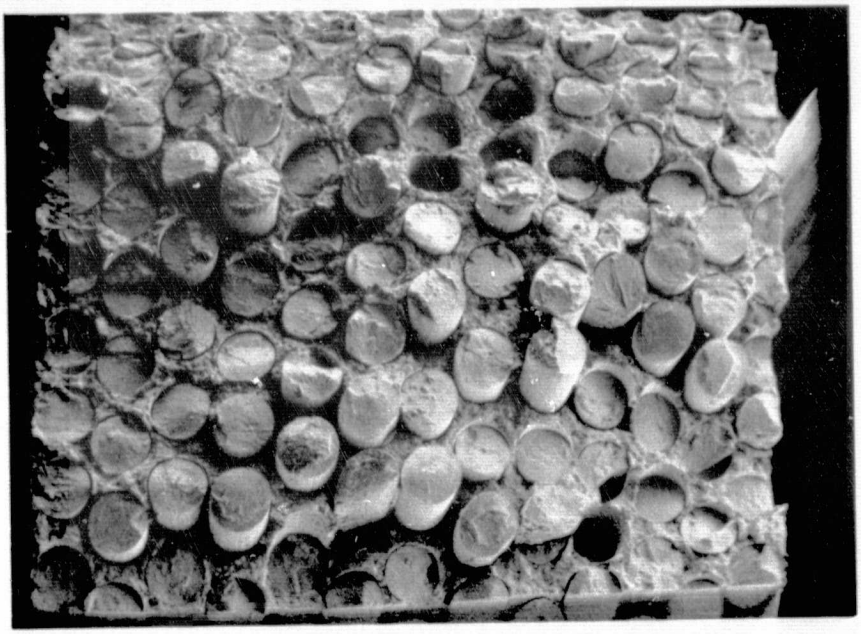
The scanning electron micrographs (Figures 22-25) of the fracture surfaces provide more insight into the understanding of the fracture processes. Brittle cleavage fractures in the tungsten filaments were observed at room temperatures (Figures 22 and 23) and also at 149°C (300°F). At this relatively high volume fraction reinforcement level, the matrix condition did not appear to significantly affect the fracture, since the Ni/1 alloy matrix was clearly not fully consolidated compared to the Ni/2 alloy matrix. At 371°C (700°F), ductile failure of the tungsten filaments was evident with cup-cone fractures and large reductions in area (Figures 24 and 25). Significantly more filament pull-out was observed with the Ni/2 alloy matrix system. It should be noted that since the 371°C specimens did not fracture completely through the cross section, they were bent at room temperature to effect failure in the last 2-3 filament rows, in order to observe the fracture surfaces. The amount of pull-out visible in Figure 24 was observable prior to this procedure. The impact-energy versus temperature data are plotted in Figure 26 with SEM fractographs for the Ni/2 matrix alloy system superimposed. The very large spread in impact energy at 371°C for the Ni/2 system was probably a function of the filament pull-out since the matrix did not appear to change role with temperature.

Static Air Oxidation

Thermogravimetric tests on specimens with 65v/o packing density cores and with the Ni/1 and NI/2 matrices were conducted at the Institute for Research Inst. (Houston) Testing Laboratories. Approximate specimen dimensions were 60mm x 12mm x 4mm. Extra matrix material was required at one end so that a small hole could be drilled (without exposing the fibers) to suspend the specimens inside the test furnace. Specimens were heated to 1093°C in a temperature programmed furnace and in a static air environment for 20 hours and 100 hours total exposure.

The weight gain data are presented in Figure 27 for both 20-hour and 100-hour exposures. As would be expected from the alloy compositions, the Ni/2 matrix system (NiCrAlY) had the lowest weight gain (better by a factor of four). In both cases, oxidation appeared to follow a parabolic relationship up to about 20 hours after which there was no significant weight change up to the maximum test time of 100 hours. The 20-hour W-Ni/1 test specimen showed an accelerated weight gain after about 10 hours, and had a total weight gain of 2.03 mg/cm^2 compared to about 1.3 mg/cm^2 (20 hours) for a duplicate sample. Visual examination indicated that an individual fiber had become exposed when the small hole, machined for suspension purposes, was prepared. Except for a slight peak at about 25 hours in the W-Ni/2 system, the weight change data did not indicate any significant spalling. Total weight gains after 100 hours were .43 and 1.54 mg/cm^2 for the Ni/2 and Ni/1 matrix systems, respectively.

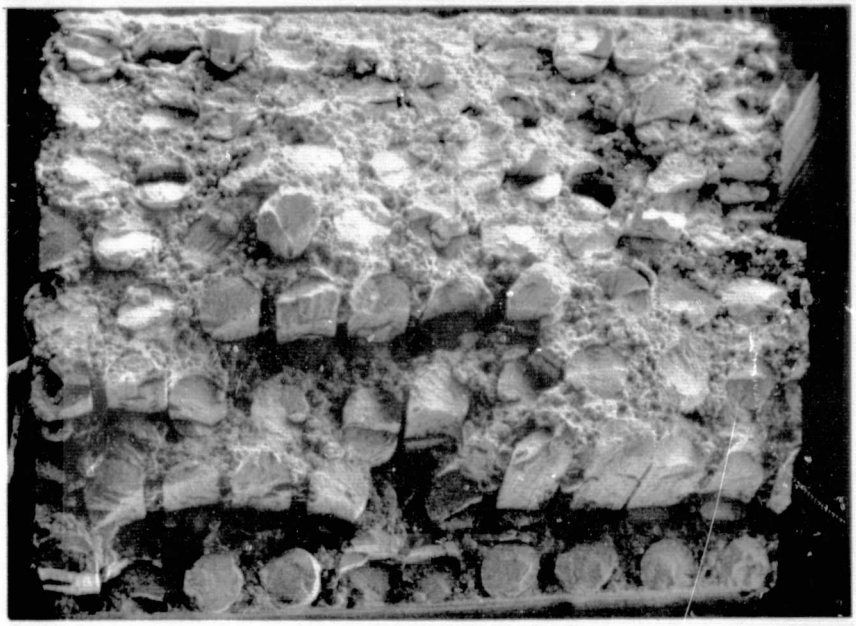
The planned metallographic analyses were not performed on these samples in time to include in this report, since the specimens were returned too late. It is planned to report these data in the follow-on Contract NAS 3-20084.



Note Brittle Cleavage Fracture in W Filaments. 20X

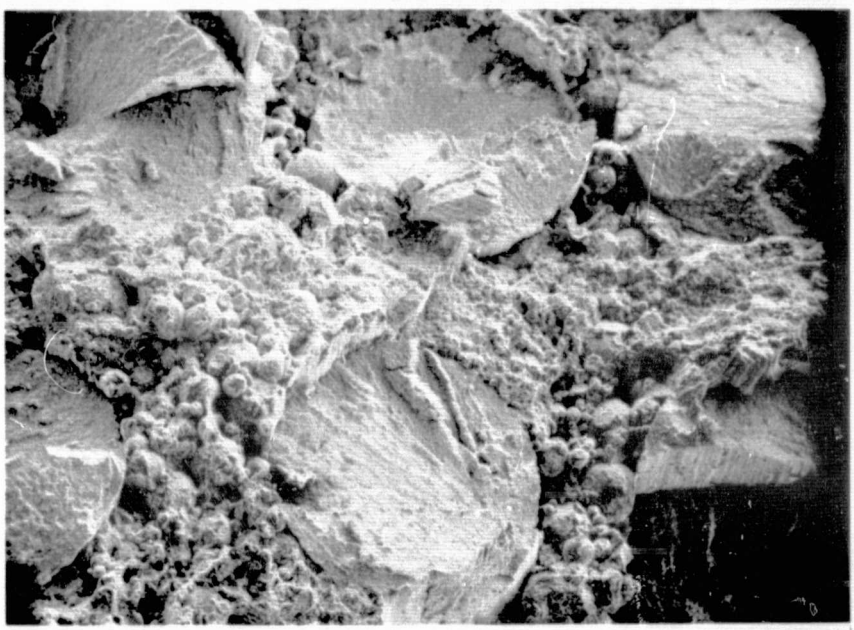
100X

Figure 22. Fracture Surface of 50v/o W-Ni/2 Miniature Izod Impact Specimen. Tested at Room Temperature.



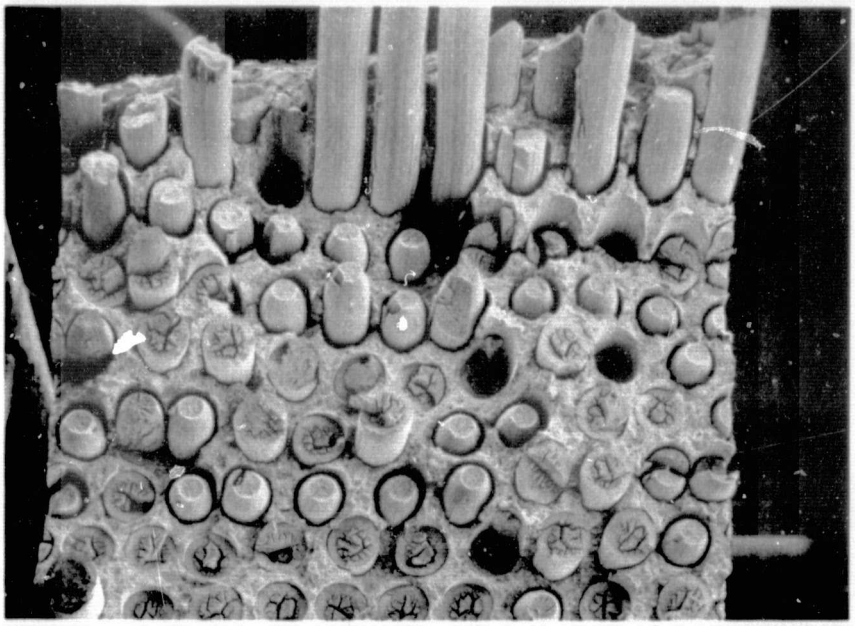
20X

Note Presence of Identifiable Powder Particles Indicating Incomplete Consolidation.

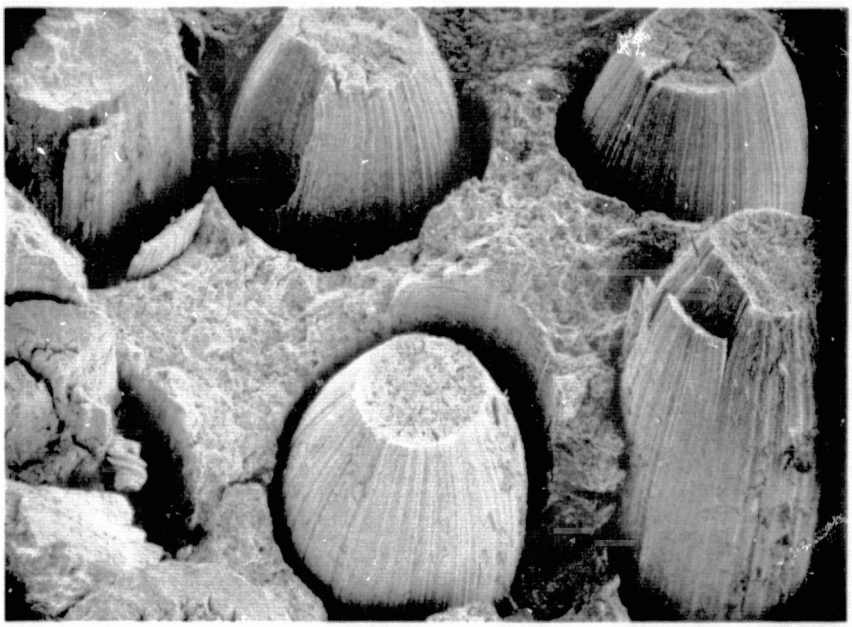


100X

Figure 23. Fracture Surface of 50v/o W-Ni/1 Miniature Izod Impact Specimen Tested At Room Temperature.



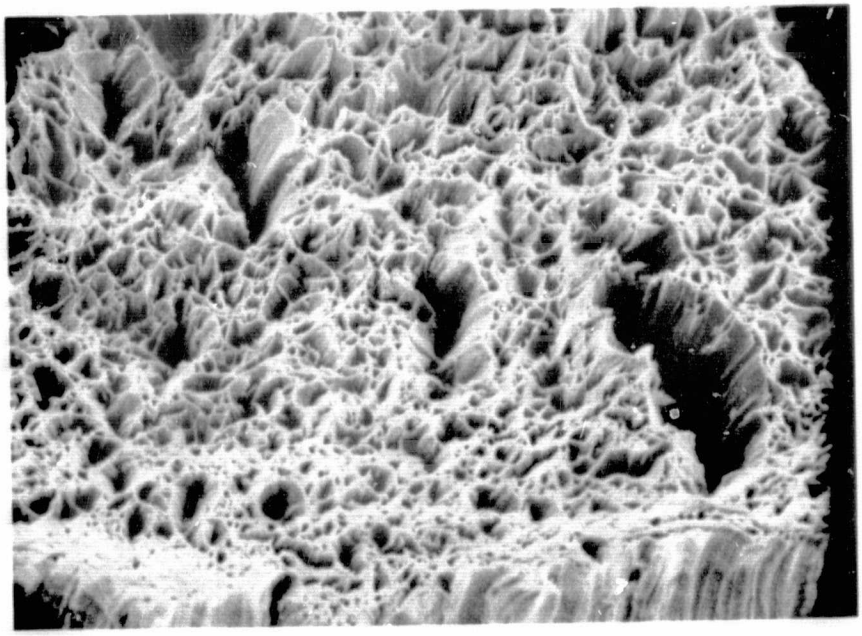
Note Ductile Filament Failure 20X



100X

Figure 24. Fracture Surface of 50v/o W-Ni/2 Miniature Izod Impact Specimen Tested at 371°C (700°F).

50v/o W-Ni/1 Ductile Fiber Failure
Poorly Consolidated Matrix 100X



Ductile Fracture Surface of Tungsten Wire 2000X

Figure 25. Fracture Surface of 50v/o W-Ni/2 Miniature Izod impact Specimen Tested at 371°C (700°F).

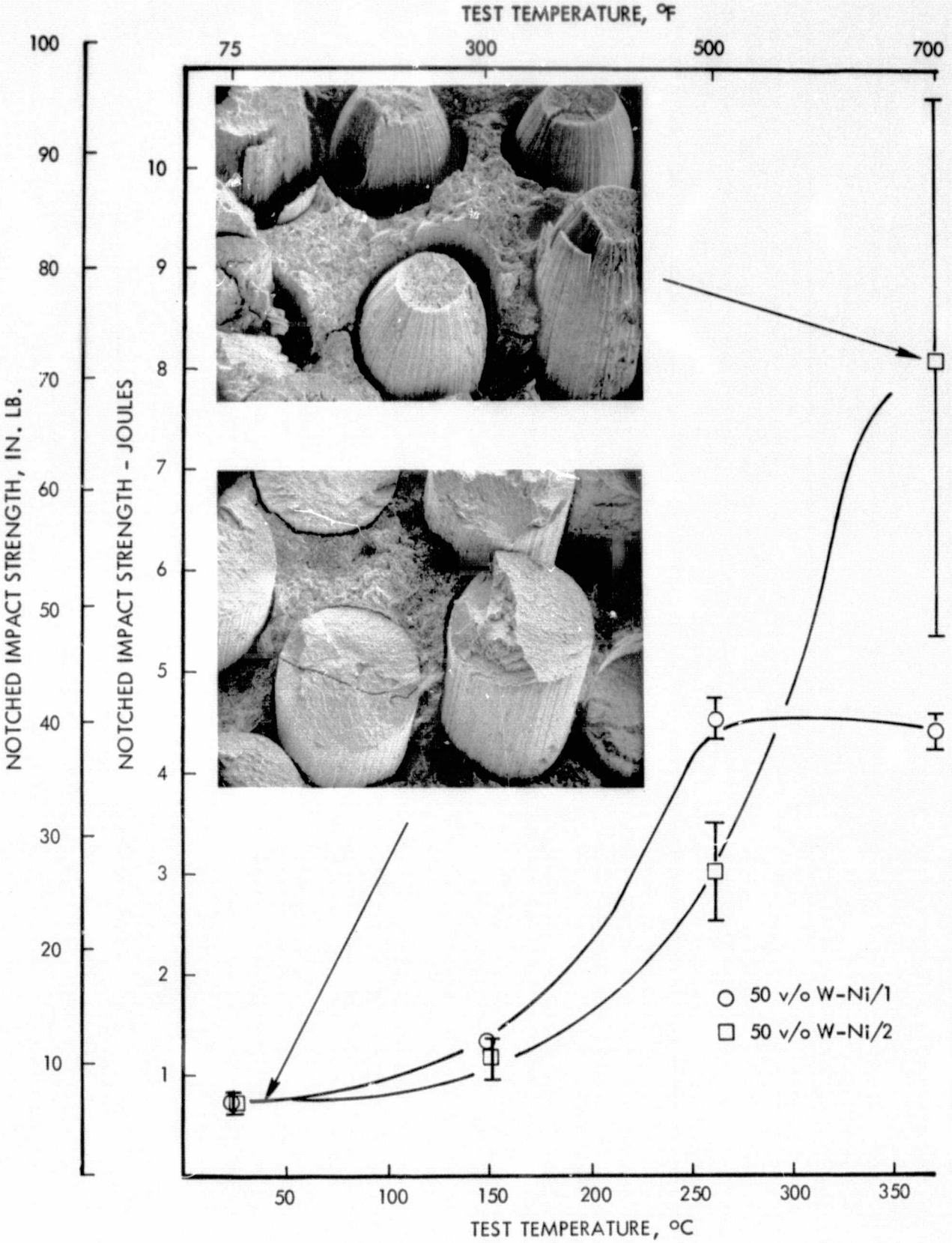


Figure 26. Impact Energy vs. Temperature for W-Superalloy Composites

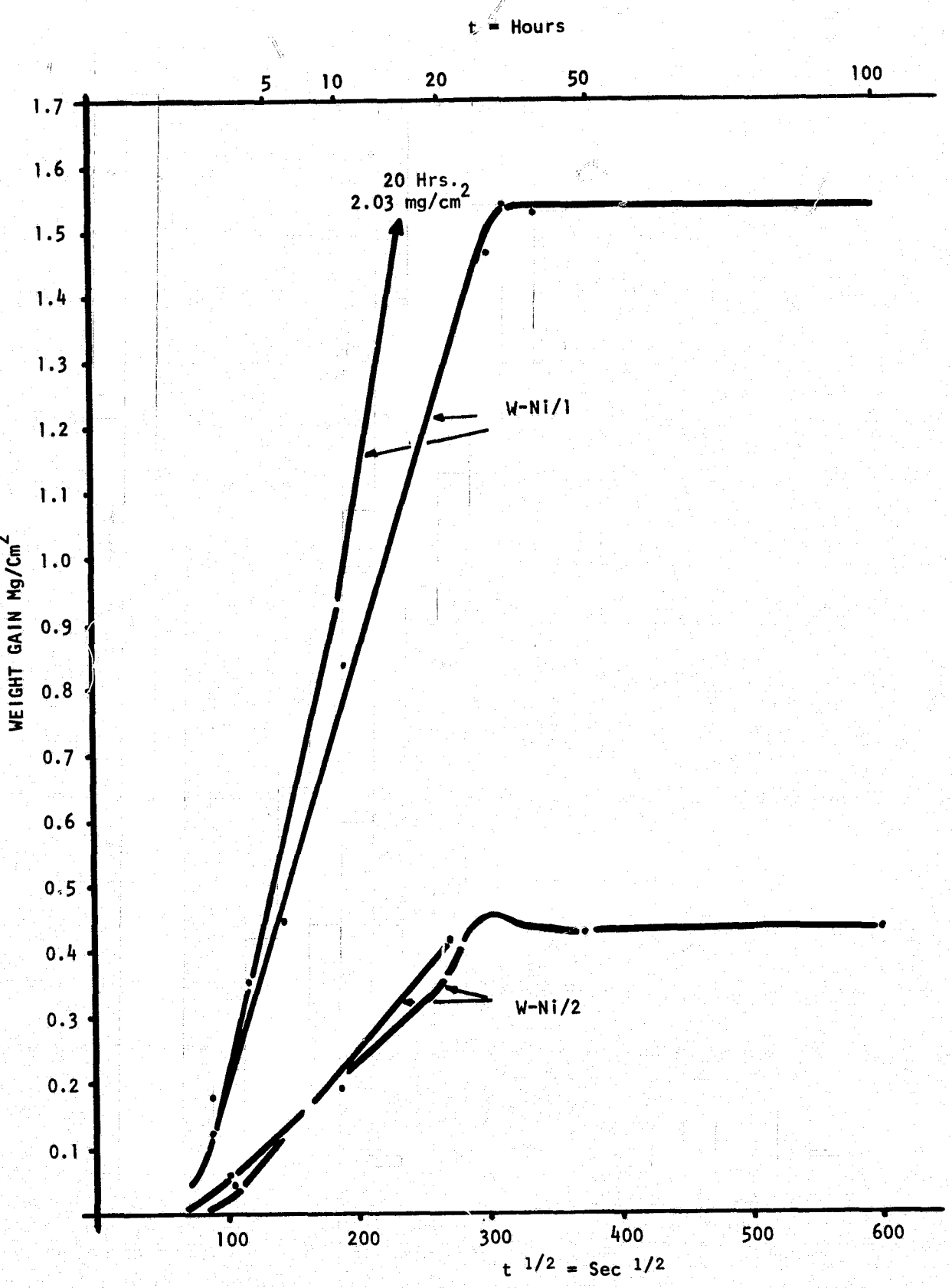


Figure 27. Static Oxidation Test Data of 1093°C (2000°F) on W-Ni/1 and W-Ni/2 Composite Systems.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The program objective was to continue development of nickel-base alloy-refractory wire reinforced composites for intended application in advanced aircraft gas turbine engines as components such as turbine blades.

A major part of the investigation was concerned with response of the selected systems to thermal cycling (thermal fatigue). Based on preliminary screening studies, two systems were selected for extended thermal fatigue cycle tests and for property evaluations which included air environment stress rupture, impact tests and static air oxidation. The major conclusions from these evaluations are as follows:

1. Multiple thermal cycling can cause a number of effects in reinforced superalloy composites which include: dimensional instability (negative creep), warpage, delamination, matrix debonding and matrix microcracking.

2. Available data indicate that these effects may be controlled by proper selection of matrix strength, fiber strength and amount and distribution of reinforcement.

3. The use of non-optimized fabrication methods was indicated to be a significant factor in many of the observed delamination and debonding failures.

4. Specimens fabricated with the NiCrAlY (powder) matrix had the best thermal fatigue resistance, surviving 1000 cycles with macroscopic failure. Internal microcracking (apparently related to inadequate bonding) did occur, however. In this respect, the 50v/o W-Ni/2 system was not as good as the 45v/o W-1ThO_2/FeCrAlY system which survived 1000 cycles 20°-1204°C (70° - 2200°F) without any structural or mechanical degradations[9].

5. The 100-hour stress rupture strengths of both 218 CS and W-Hf-C fiber reinforced NiCrAlY composites were in agreement with, or slightly higher than theoretical predictions based on published data for these fiber materials. The 1093°C - 100-hour rupture strength of about 365 MN/m^2 (53 ksi) determined for the 30v/o W-Hf-C/NiCrAlY system is impressive and translates to a specific strength advantage of about 150 percent over that of typical directionally solidified eutectics.

6. Notched impact tests (miniature Izod) indicated a ductile brittle transition temperature range between 150 and 300°C below which fracture was matrix controlled and above which fracture was controlled by the fibers.

7. At temperatures below the DBTT, the notched impact values were in the range defined as adequate (Winsa and Petrasek) for turbine blade applications, and could be improved by (a) matrix conditioning, (b) use of a tougher matrix alloy, and (c) using a lower volume fraction of reinforcement (e.g., 30v/o W-Hf-C). At temperatures above 250°C, the impact strengths are above proposed standards.

8. As would be anticipated, the static air oxidation rates at 1093°C (2000°F) of these composite systems are a function only of the matrix alloy composition.

Recommendations for further investigations on these promising systems are:

1. Design a composite system with an optimum combination of strength and thermal fatigue resistance. This is to be achieved through optimization of matrix alloy composition, fabrication parameters and fiber volume fraction and distribution. New alloy compositions may be formulated for this specific application.

2. Conduct a detailed property characterization of the selected composite system.

3. Demonstrate processibility by fabrication and destructure evaluation of airfoil shapes.

6.0 REFERENCES

1. D. W. Petrasek, R. A. Signorelli and J. W. Weeton, "Refractory Metal Fiber Nickel Alloy Composites for Use at High Temperatures", NASA TN D4787, 1968.

2. D. W. Petrasek and R. A. Signorelli, "Preliminary Evaluation of Tungsten Alloy Fiber-Nickel-Base Alloy Composites for Turbojet Engine Applications", NASA TN D-5575, February 1970.

3. E. A. Winsa and D. W. Petrasek, "Factors Affecting Miniature Izod Strength of Tungsten-Fiber-Metal-Matrix Composites, NASA TN D-7393, October 1973.

4. D. W. Petrasek and R. A. Signorelli, "Stress-Rupture Strength and Microstructural Stability of Tungsten-Hafnium-Carbon-Wire-Reinforced Superalloy Composites", NASA TN D-7773, October 1974.

5. "Fabrication of Tungsten Wire Nickel-Base Alloy Composites", Contract NAS 3-16756 and Contract NAS 3-17816, 1973-1975.

6. E. M. Breinan et al, "The Effect of Thermal Cycling on High Temperature Eutectic Composites", NMAB 308-11, January 1973.

7. W. D. Brentnall, G. D. Menke and I. J. Toth, "Creep Formability of Metallic Composites", Presented at AIME Metal Show, Detroit, October 1971.

8. R. Erturk, "Forging of Metal Matrix Composites Forming Limit", Ph.D. Thesis, 1975.

9. W. D. Brentnall et al, "Metal Matrix Composites For High Temperature Turbine Blades", Third Quarterly Progress Report on Contract N62269-75-C-0019, October 1975.

10. W. D. Brentnall et al, "Metal Matrix Composites For High Temperature Turbine Blades", Final Report on Contract N00019-74-C-0122, Mar. 1975.

cyclops CORPORATION

UNIVERSAL-CYCLOPS SPECIALTY STEEL DIVISION

TEST REPORT

Mill Order No. _____ Shipping Memo _____ Date Shipped _____

Customer Order No. 88-359-066 Specification —

Customer: TRW, Incorporated
23555 Euclid Avenue
Cleveland, Ohio 44117

Grade Unitemp AF 2-1DA

Description: Sheet and Plate

ATTEN:

CHEMICAL ANALYSIS

HEAT NO.	ITEM	C	MN	SI	S	P	CR	W	V TA	NI	MO	CO	FE	AL	TI
K70162K11	1	.34	.02	.04	.003	.001	12.16	6.15	1.65	Bal.	3.02	9.96	.35	4.57	3.02
												ZR	B		
C15345K11	2	.30	.01	.03	.002	.002	12.05	6.0	1.56	Bal.	2.96	.12 9.97	.014 .09	4.63	2.97
												ZR	B		
K70162K11	3	Same as Item #1										.11	.016		
C15345K11	4	Same as Item #2													

MECHANICAL PROPERTIES

ITEM	CONDITION	HARDNESS	YIELD	TENSILE	% EL.	% RA.	BENDS	PIECES	SIZE/WEIGHT
1*	Fully Annealed & Descaled							1	Item #1 - 22 lbs. 0.240/0.250x RW x RL
2&3**	Partial Anneal & Descaled							1	Item #2 - 10 lbs. 0.157/0.165* x RL x RW
								2	Item #3 - 10 lbs. 0.016/0.020 x RW x RL
4***								1	Item #4 - 27 lbs. 0.118/0.[illegible] x RW x RL

ITEM	GRAIN SIZE	HARDENABILITY	TREATMENT	STRESS RUPTURE VALUES STRESS - PSI	TEMP. °F	TIME - HRS.	% EL.
			*2150°F/30 min/FC to 1950°F/ 15 min/WQ (Full Anneal) **1900°F/15 min/WQ (superplastic)				

ITEM	OTHER PROPERTIES	REMARKS:

cyclops CORPORATION

UNIVERSAL-CYCLOPS SPECIALTY STEEL DIVISION
PITTSBURGH PLANT

I CERTIFY THAT THIS IS A TRUE AND CORRECT COPY OF THE TESTS SHOWN ON OUR LABORATORY RECORDS.

BY _____
AUTHORIZED SIGNATURE

0-5

7.0 APPENDIX

Vendor Certification of Alloy 2-1DA